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FINAL SAMPLING AND ANALYSIS PLAN FOR UNEXPLODED ORDNANCE SITE 1 (UXO 1)  
AND UNEXPLODED ORDNANCE SITE 2 (UXO 2) FORMERLY AREA OF CONCERN 1 (AOC  
1) MILLINGTON SUPPACT TN  
12/1/2012  
RESOLUTION CONSULTANTS

**FINAL  
SAMPLING AND ANALYSIS PLAN**

**UXO 1 and UXO 2 (formerly AOC 1)  
NAVAL SUPPORT ACTIVITY MID-SOUTH  
MILLINGTON, TENNESSEE**

**Version Number: 0**

**Prepared For:**



**Department of the Navy  
Naval Facilities Engineering Command Midwest  
201 Decatur Avenue, Building IA  
Great Lakes, Illinois 60088**

**Prepared By:**



**Resolution Consultants  
*A Joint Venture of AECOM & EnSafe*  
1500 Wells Fargo Building  
440 Monticello Avenue  
Norfolk, Virginia 23510**

**Contract Number: N62470-11-D-8013  
CTO F271**

**December 2012**

**SAP WORKSHEET #1: TITLE AND APPROVAL PAGE**  
*(UFP-QAPP Manual Section 2.1)*

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UXO 1 AND UXO 2 (Formerly AOC 1)  
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Tina Cantwell, Resolution Consultants  
Project Chemist/QA Officer/Date

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Jan Nielsen, NAVFAC Atlantic  
Quality Assurance Officer/Chemist/Date

---

Ben Brantley, Resolution Consultants  
Task Order Manager/Date

---

Ben Simes, NAVFAC Midwest  
Remedial Project Manager/Date

**SAMPLING AND ANALYSIS PLAN**  
**SAP WORKSHEET #1: TITLE AND APPROVAL PAGE**  
*(UFP-QAPP Manual Section 2.1)*  
**for**

**AREA OF CONCERN 1 AND UXO 1**  
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**1069943540**

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ou=USN, cn=NIELSEN.JANICE.L.1069943540  
Date: 2012.10.25 16:38:46 -04'00'

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\_\_\_\_\_  
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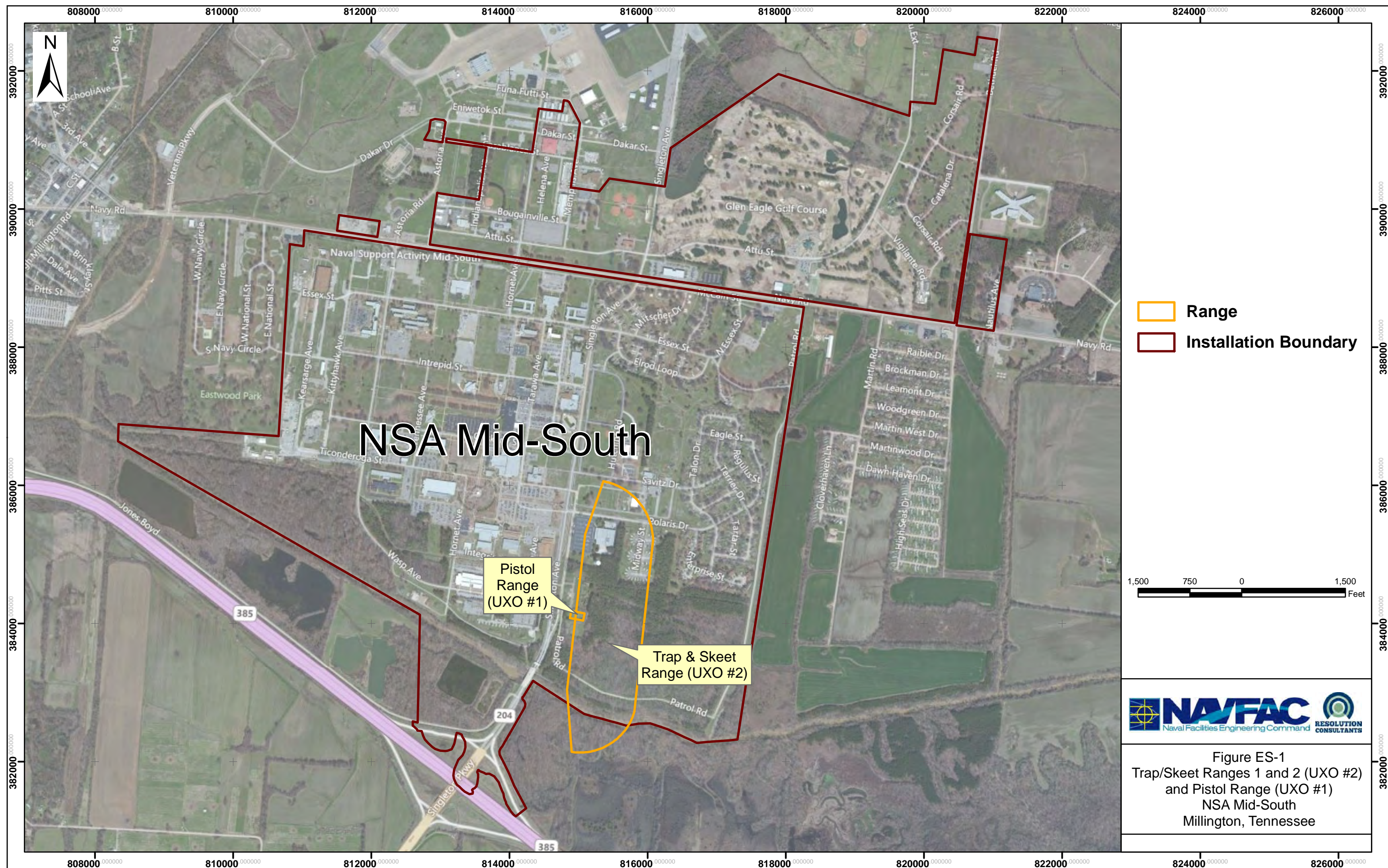
## EXECUTIVE SUMMARY

Resolution Consultants has prepared this Sampling and Analysis Plan (SAP) (Field Sampling Plan (FSP) and Quality Assurance Project Plan (QAPP) for conducting a Resource Conservation Recovery Act (RCRA) Facility Investigation at Unexploded Ordnance (UXO) 1 — Pistol Range 1 and UXO 2 — Trap/Skeet Ranges #1 and #2 (formerly known as Area of Concern 1). Site Inspections (SI) conducted in 2010 (Tt, 2010) identified lead and polynuclear aromatic hydrocarbon (PAH) concentrations above human health screening values in surface soil and to a lesser degree in subsurface soil. The objectives of the RCRA Facility Investigation (RFI) activities proposed in this SAP are to (1) fill data gaps from the SI (Tt, 2010), (2) evaluate the potential human health risk associated with impacted site media, and (3) collect adequate data for evaluating remedial alternatives that are protective of human health and the environment. The work specified in this SAP is required pursuant to NSA Mid-South's Hazardous Waste Management, RCRA Corrective Action Permit (TN2 17 002 2600, TNHW-149).

Naval Support Activity (NSA) Mid-South is in Millington, Shelby County, Tennessee, approximately 20 miles north of Memphis and 7 miles east of the Mississippi River. It encompasses 1,600 acres and serves as one of the largest inland U.S. Navy installations in the country. As of April 2012, the base employed 7,500 people including enlisted, officer, and civilian personnel. Trap/Skeet Ranges #1 and #2 consist of approximately 79 acres near the southern border of the installation. The skeet ranges were built around 1943, with Trap/Skeet Range #1 having an unrecorded closure date sometime after World War II and Trap/Skeet Range #2 closing in 2005. The former Pistol Range 1 was constructed within Trap/Skeet Ranges #1 and #2 and consists of approximately 0.5 acre from the firing line to a containment berm. The pistol range was constructed after 1948 and remained in use until 1994. Figure ES-1 shows the two sites within NSA Mid-South. No remedial efforts are documented at either of the two sites.

The Department of Defense (DoD) has established a separate program, known as the Munitions Response Program (MRP), to address closed military ranges. A Preliminary Assessment (Malcolm-Pirnie, Inc., 2005) identified seven Navy MRP-eligible sites at NSA Mid-South that were recommended for an SI as a result of the Navy's munitions training activities, including UXO 1 and UXO 2. The Munitions Constituents (MC) detected in the SI consisted of lead in surface soil at UXO 2 and UXO 1, and PAHs in soil at UXO 2. The term Munitions and Explosives of Concern (MEC), applies to Discarded Military Munitions DMM), UXO, and MC that have been found at high enough concentrations to pose an explosive hazard. MEC is not suspected at UXO 2; however, all work conducted at UXO 1 will be under the supervision of a UXO technician because of the possible presence of unexpended small arms ammunition.







The information provided in the worksheets in this SAP was developed based on the results of the October 2012 project scoping meeting attended by the Navy's Base Cleanup Team (BCT) which consists of representatives from the Navy, United States Geological Survey (USGS), Tennessee Department of Environment and Conservation (TDEC), and Resolution Consultants (see Worksheet #9 for attendees). Worksheet #10 contains the problem statement and general Conceptual Site Models (CSMs) for the skeet ranges and small arms range. The CSMs were used as the basis for the development of the Data Quality Objectives (DQOs), which are listed in Worksheet #11. The remaining worksheets describe the RFI sampling, analytical, and data evaluation procedures, including data-quality requirements.

Standard operating procedures (SOPs) for all field procedures and data collection are in Appendix A. Field forms to be used for this project are in Appendix B. Appendices C and D provide laboratory accreditation certification and SOPs, respectively.



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Appendix A	Standard Operating Procedures
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## Acronyms and Abbreviations

AOC	Area of Concern
BCT	Base Cleanup Team
bgs	Below Ground Surface
BRAC	Base Realignment and Closure Program
C	Celsius
CAS	Chemical Abstracts Service
CCC	Calibration check compound
CCV	Continuing Calibration Verification
CLEAN	Comprehensive Long-Term Environmental Action Navy
COD	Coefficient of determination
COPC	Contaminant of Potential Concern
CSM	Conceptual Site Model
CTO	Contract Task Order
CV	Calibration Verification
DDT	4,4'-dichlorodiphenyltrichloroethane
DoD	Department of Defense
DO	Dissolved Oxygen
DQO	Data Quality Objective
DSWM	Division of Solid Waste Management
EB	Equipment blank
EICP	Extracted ion current profile
ELAP	Environmental Laboratory Accreditation Program
FD	Field duplicate
FTL	Field Team Leader
GC	Gas Chromatography
GCAL	Gulf Coast Analytical Laboratory
GC/MS	Gas Chromatograph/Mass Spectrometer
GC/ECD	Gas Chromatograph/Electron Capture Detector
HSO	Health and Safety Officer
ICAL	Initial Calibration
ICP/MS	Inductively Coupled Plasma Spectroscopy/Mass Spectrometer
ICV	Initial calibration verification
ID	Identification
IDW	Investigative Derived Waste
IR	Installation Restoration

LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOD	Limit of Detection
LOQ	Limit of Quantification
MC	Munitions Constituents
MCL	Maximum Contaminant Level
MS/MSD	Matrix Spike/Matrix Spike Duplicate
MEC	Munitions and Explosives of Concern
MRP	Munitions Response Program
mg/kg	Milligrams per kilogram
mV	Millivolt
NA	Not Applicable
NAVFAC	Naval Facilities Engineering Command
NAVFAC Midwest	Naval Facilities Engineering Command Midwest Division
NIRIS	Naval Installation Restoration Information Solution
NSA	Naval Support Activity
ORP	Oxidation-Reduction Potential
%	Percent
%D	Percent Difference
%R	Percent Recovery
%RSD	Percent Relative Standard Deviation
PAH	Polynuclear aromatic hydrocarbons
PAL	Project Action Level
PDF	Portable document format
PM	Project Manager
POC	Point of Contact
PVC	Polyvinyl chloride
QA	Quality Assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
QSM	Quality Systems Manual
r <sup>2</sup>	Correlation coefficient
RCRA	Resource Conservation and Recovery Act
RF	Response factors
RFI	Resource Conservation and Recovery Act Facility Investigation
RL	Reporting limit
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RRT	Relative retention times
RSL	Regional Screening Level



SAP	Sampling and Analysis Plan
SI	Site Inspection
SIM	Selective ion monitoring
SOP	Standard Operating Procedure
SSL	Soil Screening Level
SSO	Site Safety Officer
SWMU	Solid Waste Management Unit
TBD	To Be Determined
TDEC	Tennessee Department of Environment and Conservation
TOM	Task Order Manager
UFP	Uniform Federal Policy
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey
UXO	Unexploded Ordnance



**SAP WORKSHEET #2: SAMPLING AND ANALYSIS PLAN IDENTIFYING INFORMATION**

*(UFP-QAPP Manual Section 2.2.4)*

**Site Name/Number:** UXO 2 & UXO 1  
NSA Mid-South; Millington, Tennessee

**Operable Unit:** Not Applicable (NA)

**Contractor Name:** Resolution Consultants

**Contract Number:** N62470-11-D-8013

**Contract Title:** Comprehensive Long-term Environmental Action (CLEAN)

**Work Assignment No.:** Contract Task Order (CTO) F271

1. **This sampling and analysis plan (SAP) was prepared in accordance with the requirements of:** *Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP) (USEPA, 2005), U.S. Environmental Protection Agency (USEPA) Guidance for Quality Assurance Project Plans, EPA QA/G-5 (USEPA, 2002), and Guidance on Systematic Planning Using the Data Quality Objective Process (USEPA, 2006).*
2. **Identify regulatory program:**  
RCRA
3. **This SAP is a project specific SAP.**
4. **List organizational partners (stakeholders) and connection with lead organizations:**

Organization Partners/Stakeholders	Connection
TDEC Division of Solid Waste Management (TDEC DSWM)	Lead Regulatory Oversight
Naval Facilities Engineering Command Midwest (NAVFAC Midwest)	Lead Agency
NSA Mid-South	Property Owner
USGS	Base Clean-up Team Partner
Resolution Consultants	Base Clean-up Team Partner



5.     **Lead Agency:** NAVFAC Midwest
  
6.     **If any required SAP elements and required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below:** NA, as there are no exclusions.



### SAP WORKSHEET #3: DISTRIBUTION LIST

(UFP-QAPP Manual Section 2.3.2)

Distribution List				
SAP Recipients	Title	Organization	Telephone Number	E-mail Address or Mailing Address
Ben Simes	Naval Remedial Project Manager (RPM)	NAVFAC Midwest 201 Decatur Avenue, Building 1A Great Lakes, IL 60088-2801	(847) 688-2600 x 320	benjamin.simes@navy.mil
Howard Hickey	Naval Remedial Project Manager (co-RPM)	NAVFAC Midwest 201 Decatur Avenue, Building 1A Great Lakes, IL 60088-2801	(847) 688-5999 x 243	howard.hickey@navy.mil
Jim Heide	Head of Public Works/Environmental	NSA Mid-South — Bldg. 455 5722 Integrity Drive Millington, TN 38054-5000	(901) 874-5367	jim.heide@navy.mil
Roger Donovan	TDEC DSWM Project Manager	TDEC — DSWM 5 <sup>th</sup> Floor, L&C Annex 401 Church St. Nashville, TN 37243-1538	(615) 532-0864	roger.donovan@tn.gov
Charlie Burroughs	TDEC DSWM Division Head	TDEC — DSWM 5 <sup>th</sup> Floor, L&C Annex 401 Church St. Nashville, TN 37243-1538	(615) 532-0863	charles.burroughs@tn.gov
Ben Brantley	Task Order Manager (TOM)	Resolution Consultants 5724 Summer Trees Drive Memphis, TN 38134	(901) 372-7962	bbrantley@ensafe.com
Claire Barnett	Project Engineer/co-TOM	Resolution Consultants 5724 Summer Trees Drive Memphis, TN 38134	(901) 372-7962	cbarnett@ensafe.com
Tina Cantwell	Project Chemist/Data Manager/Quality Assurance Officer (QAO)	Resolution Consultants 5724 Summer Trees Drive Memphis, TN 38134	(901) 372-7962	tcantwell@ensafe.com
Corey Coleman	Field Team Leader (FTL)	Resolution Consultants 5724 Summer Trees Drive Memphis, TN 38134	(901) 372-7962	ccoleman@ensafe.com





Distribution List				
SAP Recipients	Title	Organization	Telephone Number	E-mail Address or Mailing Address
Shelley Bourgeois	Laboratory Project Manager	Gulf Coast Analytical Laboratories 7979 GSRI Road Baton Rouge, LA 70820	(225) 214-7077	shelley.bourgeois@gcal.com

**Notes:**

Each person listed in this table will be responsible for distributing copies of this SAP to appropriate personnel within their organization.

NAVFAC Midwest = Department of the Navy, Naval Facilities Engineering Command, Midwest

NSA = Naval Support Activity

TDEC DSWM = Tennessee Department of Environment and Conservation Division of Solid Waste Management



## SAP WORKSHEET #4: PROJECT PERSONNEL SIGN-OFF SHEET

(UFP-QAPP Manual Section 2.3.2)

Project Personnel Sign-Off Sheet					
Name	Organization/Title/Role	Telephone Number	Signature/e-mail receipt	SAP Section Reviewed	Date SAP Read
<b>Navy and Regulator Project Team Personnel</b>					
Ben Simes	NAVFAC Midwest/RPM	(847) 688-2600 x 320		All	
Howard Hickey	NAVFAC Midwest/RPM	(847) 688-5999 x 243		All	
Roger Donovan	TDEC DSWM/RPM	(615) 532-0864		All	
Charlie Burroughs	TDEC DSWM/Division Head	(615) 532-0863		All	
<b>Resolution Consultants Project Team Personnel</b>					
Ben Brantley	Resolution Consultants/TOM	(901) 372-7962		All	
Claire Barnett	Resolution Consultants/Co-TOM	(901) 372-7962		All	
Tina Cantwell	Resolution Consultants/Chemist/QAO/ata Manager	(901) 372-7962		All	
Corey Coleman	Resolution Consultants/FTL	(901) 372-7962		All	
<b>Subcontractor Personnel</b>					
Shelley Bourgeois	Gulf Coast Analytical Laboratories/Laboratory Project Manager	(225) 214-7077		Worksheets #6, #12, #14, #15, #19, #20, #23-28, #30, and #34-36	

### Notes:

Persons listed on this worksheet will be responsible for distributing the SAP to the appropriate people within their organizations.

NAVFAC Midwest = Department of the Navy, Naval Facilities Engineering Command, Midwest  
TDEC DSWM = Tennessee Department of Environment and Conservation Division of Solid Waste Management  
RPM = Remedial Project Manager  
TOM = Task Order Manager  
QAO = Quality Assurance Officer  
FTL = Field Team Leader

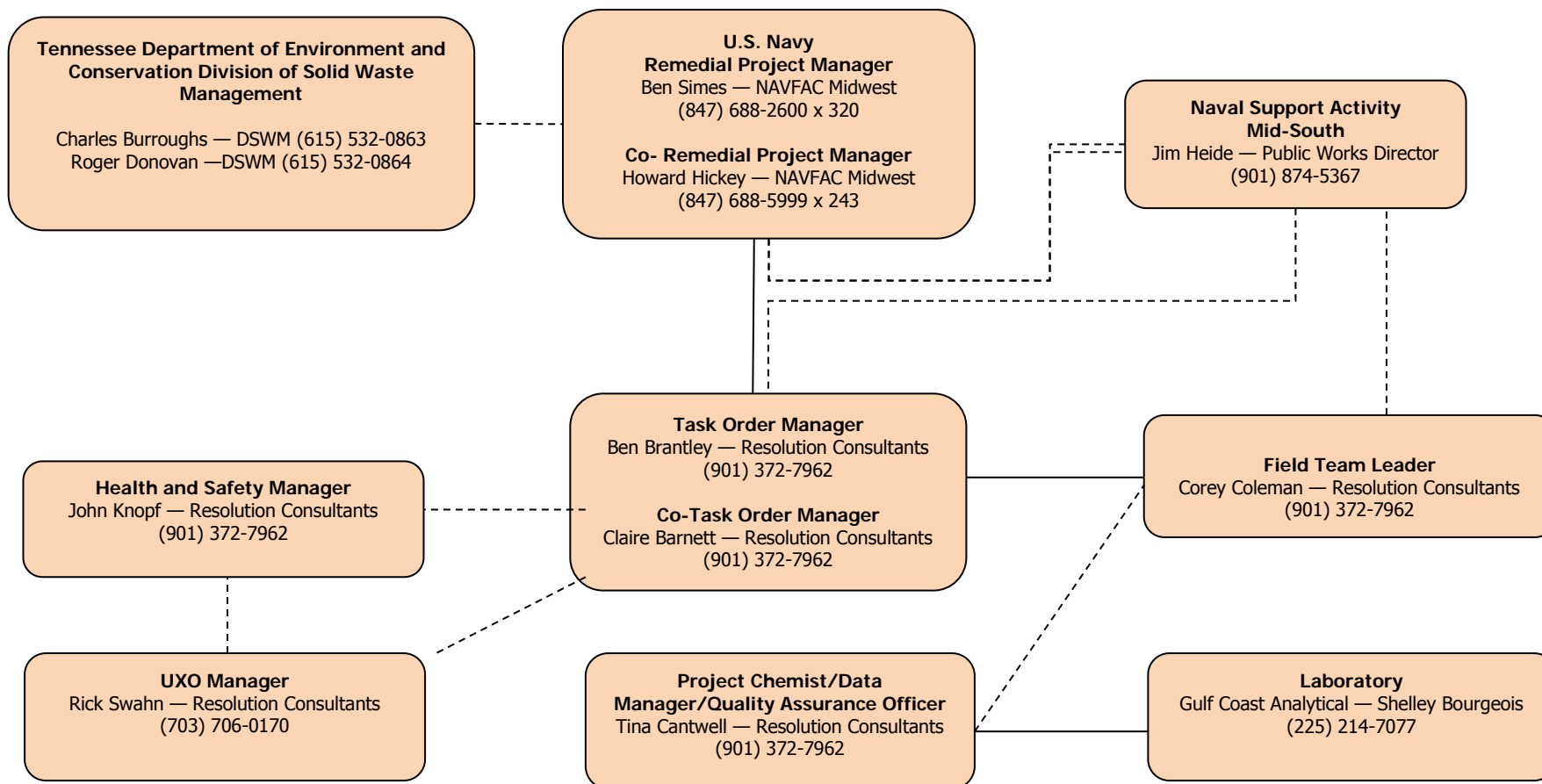


## SAP WORKSHEET #5: PROJECT ORGANIZATIONAL CHART

(UFP-QAPP Manual Section 2.4.1)

Lines of Authority —————

Lines of Communication - - - - -





## SAP WORKSHEET #6: COMMUNICATION PATHWAYS

*(UFP-QAPP Manual Section 2.4.2)*

The communication pathways for the SAP are shown below.

Communication Pathways				
Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
Regulatory Agency Interface	Navy RPM Navy Co-RPM	Ben Simes Howard Hickey	(847) 688-2600 x 320 (847) 688-5999 x 243	The Navy RPM informs regulatory agency of work progress on a periodic basis.
Progress Reports	Resolution Consultants FTL Resolution Consultants TOM Navy RPM	Corey Coleman Ben Brantley Ben Simes	(901) 372-7962 (901) 372-7962 (847) 688-2600 x 320	FTL verbally informs the TOM on a daily basis — field updates. TOM provides a weekly update to the RPM either by phone message and/or e-mail each Friday afternoon field activities are taking place.
Gaining Site Access	Resolution Consultants FTL NSA Mid-South Head of Public Works	Corey Coleman Jim Heide	(901) 372-7962 (901) 874-5367	The Resolution Consultants FTL will contact the NSA Mid-South point of contact verbally or via e-mail at least 3 days before commencement of field work to arrange for access to the site for all field personnel.
Obtaining Utility Clearances for Intrusive Activities	Resolution Consultants FTL NSA Mid-South IR Site Manager	Corey Coleman Rob Williamson	(901) 372-7962 (901) 874-5399	The Resolution Consultants FTL will coordinate verbally or via e-mail with NSA Mid-South point of contact at least 14 days in advance of site access to initiate the utility clearance process for all intrusive sampling locations.
Stop Work due to Safety Issues	Resolution Consultants FTL/SSO Resolution Consultants TOM Resolution Consultants HSO Navy IR Site Manager	Corey Coleman Ben Brantley John Knopf Rob Williamson	(901) 372-7962 (901) 372-7962 (901) 372-7962 (901) 874-5399	The responsible party verbally informs the FTL, TOM, and subcontractors within 1 hour of recommendation to stop work and within 24 hours of recommendation to restart work. Responsible party follows verbal notification with an e-mail to the Project Team within 24 hours.  If a subcontractor is the responsible party, the subcontractor PM must verbally inform the Resolution Consultants SSO within 15 minutes and the Resolution Consultants SSO will then follow the procedure listed above.
SAP Changes before Field/Laboratory work	Resolution Consultants TOM Navy RPM	Ben Brantley Ben Simes	(901) 372-7962 (847) 688-2600 x 320	Any change of the approved SAP will be made only upon authorization of the Navy RPM and regulatory agencies. The Resolution Consultants TOM is responsible for initiating any SAP change requests via the communication channels described for the Navy and regulatory agencies.





Communication Pathways				
Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
SAP Changes in the Field	Resolution Consultants FTL/SSO Resolution Consultants TOM Navy RPM	Corey Coleman Ben Brantley Ben Simes	(901) 372-7962 (901) 372-7962 (847) 688-2600 x 320	FTL informs TOM verbally within same day; TOM informs Navy RPM via e-mail within 24 hours; TOM sends a concurrence letter to RPM, if warranted, within 7 calendar days and RPM signs the letter within 5 business days of receipt. Scope change is to be implemented before work is executed. Document the change on a field task modification request form (within 2 business days) or SAP amendment (within timeframe agreed to by Project Team).
Field Corrective Actions	Resolution Consultants FTL/SSO Resolution Consultants TOM Navy RPM	Corey Coleman Ben Brantley Ben Simes	(901) 372-7962 (901) 372-7962 (847) 688-2600 x 320	FTL informs TOM verbally within same day; TOM informs Navy RPM via e-mail within 24 hours that corrective actions have been implemented. Corrective actions will be documented in weekly progress reports. Navy RPM will notify TDEC of any significant corrective actions taken.
Recommendations to stop work and initiate work upon corrective action	Resolution Consultants FTL/SSO Resolution Consultants TOM Resolution Consultants QAO Navy RPM	Corey Coleman Ben Brantley Tina Cantwell Ben Simes	(901) 372-7962 (901) 372-7962 (901) 372-7962 (847) 688-2600 x 320	Responsible party verbally informs the TOM, FTL, and subcontractors within 1 hour of recommendation to stop work and within 24 hours of recommendation to restart work. Responsible party follows verbal notification with an e-mail to the Project Team within 24 hours.
Sample Receipt and Laboratory Quality Variances	Gulf Coast Analytical Laboratory PM Resolution Consultants FTL Resolution Consultants TOM	Shelley Bourgeois Corey Coleman Ben Brantley	(225) 214-7077 (901) 372-7962 (901) 372-7962	<p>The Laboratory PM will notify (verbally or via e-mail) the Resolution Consultants FTL immediately upon receipt of any chain of custody/sample receipt variances for clarification or direction from the Resolution Consultants FTL.</p> <p>The Resolution Consultants FTL will notify (verbally or via e-mail) the Resolution Consultants TOM within 1 business day, if corrective action is required.</p> <p>The Resolution Consultants TOM will notify (verbally or via e-mail) the Laboratory PM and the Resolution Consultants FTL within 1 business day of any required corrective action.</p>
Analytical Corrective Actions	Gulf Coast Analytical Laboratory PM Resolution Consultants Project Chemist	Shelley Bourgeois Tina Cantwell	(225) 214-7077 (901) 372-7962	The laboratory shall notify the Resolution Consultants' chemist of any analytical data anomaly within 1 business day of discovery. After the laboratory receives guidance from Resolution Consultants' chemist, the laboratory shall initiate any corrective action to prevent further anomalies.



Communication Pathways				
Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
Analytical Data Quality Issues	Gulf Coast Analytical Laboratory PM Resolution Consultants Project Chemist Resolution Consultants TOM Navy RPM	Shelley Bourgeois Tina Cantwell Ben Brantley Ben Simes	(225) 214-7077 (901) 372-7962 (901) 372-7962 (847) 688-2600 x 320	<p>The laboratory PM notifies (verbally or via e-mail) the Resolution Consultants' chemist within 1 business day of when an issue related to laboratory data is discovered. Resolution Consultants' chemist notifies Resolution Consultants' TOM within 1 business day.</p> <p>Resolution Consultants' chemist notifies the Resolution Consultants' TOM verbally or via e-mail within 48 hours of validation completion that a non-routine and significant laboratory quality deficiency has been detected that could affect this project and/or other projects. Resolution Consultants' TOM verbally advises the Navy RPM within 24 hours of notification from the chemist. The Navy RPM takes corrective action that is appropriate for the identified deficiency.</p>
Reporting Data Validation Issues/Data Validation Corrective Actions	Resolution Consultants Project Chemist Resolution Consultants TOM	Tina Cantwell Ben Brantley	(901) 372-7962 (901) 372-7962	<p>The Resolution Consultants project chemist or data validator will perform validation as specified in Worksheets #34, #35, and #36, and will contact the laboratory as soon as possible if issues are found that require corrective action.</p> <p>If, during the data validation process, the Resolution Consultants project chemist or data validator identifies non-usable data that require corrective action, the Resolution Consultants TOM will coordinate with the project chemist to take corrective action appropriate for the identified deficiency to ensure the project objectives are met. Corrective action may include resampling and/or reanalyzing the affected samples, as determined by the Resolution Consultants TOM.</p>



Communication Pathways				
Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
Notification of Non-Usable Data	Gulf Coast Analytical Laboratory PM Resolution Consultants Project Chemist Resolution Consultants TOM Navy RPM TDEC RPM	Shelley Bourgeois Tina Cantwell Ben Brantley Ben Simes Roger Donovan	(225) 214-7077 (901) 372-7962 (901) 372-7962 (847) 688-2600 x 320 (615) 532-0864	<p>If the laboratory determines that any data they have generated is non-usable, the Laboratory PM will notify (verbally or via e-mail) the Resolution Consultants project chemist within 1 business day of when the issue is discovered.</p> <p>The Resolution Consultants project chemist will notify (verbally or via e-mail) the Resolution Consultants TOM within 1 business day of the need for corrective action, if the non-usable data is a significant issue (i.e., critical sample data). Corrective action may include resampling and/or reanalyzing the affected samples.</p> <p>If the Resolution Consultants project chemist or data validator identifies non-usable data during the data validation process, the TOM will be notified verbally or via e-mail within 48 hours of validation completion that a non-routine and significant laboratory quality deficiency has resulted in non-usable data.</p> <p>The Resolution Consultants TOM will take corrective action appropriate for the identified deficiency to ensure the project objectives are met. The Resolution Consultants TOM will notify (verbally or via e-mail) the Navy RPM of any problems with the laboratory or analysis that could significantly affect the usability of the data or project failures that impact the ability to complete the scope of work. The Navy RPM, may at his discretion, contact the Navy project chemist for assistance in problem resolution. Such notification will be made within 1 business day of when the issue is discovered. The Navy RPM will notify the TDEC RPM when any significant corrective action is taken.</p>

**Notes:**

RPM = Remedial Project Manager  
 TOM = Task Order Manager  
 IR = Installation Restoration  
 HSO = Health and Safety Officer  
 TDEC = Tennessee Department of Environment and Conservation  
 PM = Project Manager

FTL = Field Team Leader  
 NSA = Naval Support Activity  
 SSO = Site Safety Officer  
 SAP = Sampling and Analysis Plan  
 QAO = Quality Assurance Officer



## SAP WORKSHEET #7: PERSONNEL RESPONSIBILITIES TABLE

*(UFP-QAPP Manual Section 2.4.3)*

Name	Title/Role	Organizational Affiliation	Responsibilities
Ben Simes	Remedial Project Manager	NAVFAC Midwest	Primary Point of Contact for the Navy. Oversees project implementation, including scoping, data review, and evaluation, on behalf of the Navy.
Howard Hickey	Co-Remedial Project Manager	NAVFAC Midwest	Secondary Point of Contact for the Navy and provides support to the Navy Remedial Project Manager and may act in same roles (e.g., oversees project implementation, including scoping, data review, and evaluation, on behalf of the Navy).
Roger Donovan	Remedial Project Manager	TDEC DSWM	Functions as primary TDEC interface. Participates in scoping and data review/evaluation, and provides review and approval of project deliverables.
Charles Burroughs	Remedial Project Manager	TDEC DSWM	Functions as secondary TDEC interface. Participates in scoping and data review/evaluation, and provides review and approval of project deliverables.
Ben Brantley	Task Order Manager	Resolution Consultants	Primary point of contact for Resolution Consultants. Oversees project implementation, including financials, schedule, and technical aspects.
Claire Barnett	Co-Task Order Manager/Project Engineer	Resolution Consultants	Secondary point of contact for Resolution Consultants. Provides co-Task Order management support and head engineer for remedy evaluation.
Corey Coleman	Field Team Leader/Site Safety Officer	Resolution Consultants	Supervises, coordinates, and performs field activities. Responsible for onsite project-specific health and safety training and monitoring site conditions. Details are in Health and Safety Plan.
Tina Cantwell	Project Chemist/QAO/Data Validation Manager	Resolution Consultants	As project chemist, prepares laboratory scopes of work, and coordinates laboratory related functions with laboratory. Performs or oversees data quality reviews and quality assurance of data validation deliverables. As QAO, ensures quality aspects of the project are implemented, documented, and maintained. As data validation manager, performs or oversees data validation and data input in both the project database and the Navy's Naval Installation Restoration Information Solution database.
John Knopf	Health and Safety Officer	Resolution Consultants	Responsible for providing health and safety training for all personnel and approving the site-specific Health and Safety Plan. Prepares health and safety reports for management.
Rick Swahn	UXO Manager	Resolution Consultants	Provides technical oversight of MEC avoidance procedures





Name	Title/Role	Organizational Affiliation	Responsibilities
Shelley Bourgeois	Laboratory Project Manager/Analytical Subcontractor	Gulf Coast Analytical Laboratories	Coordinates analyses with laboratory staff, ensures that scope of work is followed, provides quality review of data packages, and communicates with Resolution Consultants project staff.

**Notes:**

TDEC DSWM = Tennessee Department of Environment and Conservation — Division of Solid Waste Management  
NAVFAC Midwest = Department of the Navy, Naval Facilities Engineering Command, Midwest  
QAO = Quality Assurance Officer  
UXO = unexploded ordnance  
MEC = munitions and explosives of concern



## SAP WORKSHEET #8: SPECIAL PERSONNEL TRAINING REQUIREMENTS TABLE

*(UFP-QAPP Manual Section 2.4.4)*

Project Function	Specialized Training By Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/Organizational Affiliation	Location of Training Records/Certificates
Provide MEC avoidance services for the field team	Basic EOD School	Department of Defense	April 1991	Explosive Ordnance Disposal Technicians	UXO Technician/Resolution Consultants UXO Technician/Resolution Consultants	On file

**Notes:**

MEC = Munitions and explosives of concern  
UXO = Unexploded ordnance



## SAP WORKSHEET #9: PROJECT PLANNING SESSION PARTICIPANTS SHEET

(UFP-QAPP Manual Section 2.5.1)

<b>Project Name:</b> NSA Mid-South <b>Project Dates of Sampling:</b> Field Activities for 2013 <b>Project Manager:</b> Ben Brantley			<b>Site Name:</b> UXO 2 & UXO 1 <b>Site Location:</b> NSA Mid-South; Millington, Tennessee	
<b>Date of Session:</b> October 10 — 11, 2012 Base Cleanup Team Meeting <b>Scoping Session Purpose:</b> Conceptual Site Model, Data Quality Objectives, and Sampling Design for UXO 2 and UXO 1				
Name	Title	Affiliation	Phone #	E-mail Address
Roger Donovan	Remedial Project Manager	TDEC DSWM	(615) 532-0864	<a href="mailto:roger.donovan@tn.gov">roger.donovan@tn.gov</a>
Charles Burroughs	Head of DSWM	TDEC DSWM	(615) 532-0863	<a href="mailto:charles.burroughs@state.tn.us">charles.burroughs@state.tn.us</a>
Jim Heide	Head of Public Works/Environmental	NSA Mid-South	(901) 874-5467	<a href="mailto:jim.heide@navy.mil">jim.heide@navy.mil</a>
Jack Carmichael	Technical Specialist	USGS	(615) 837-4704	<a href="mailto:jkcarmic@usgs.gov">jkcarmic@usgs.gov</a>
Ben Simes	Remedial Project Manager	NAVFAC Midwest	(847) 688-2600 x320	<a href="mailto:benjamin.simes@navy.mil">benjamin.simes@navy.mil</a>
Howard Hickey	Co-Remedial Project Manager	NAVFAC Midwest	(847) 688-5999 x243	<a href="mailto:howard.hickey@navy.mil">howard.hickey@navy.mil</a>
Ben Brantley	Task Order Manager	Resolution Consultants	(901) 372-7962	<a href="mailto:bbrantley@ensafe.com">bbrantley@ensafe.com</a>
Claire Barnett	Co-Task Order Manager	Resolution Consultants	(901) 372-7962	<a href="mailto:cbarnett@ensafe.com">cbarnett@ensafe.com</a>
Corey Coleman	Field Team Leader	Resolution Consultants	(901) 372-7962	<a href="mailto:ccoleman@ensafe.com">ccoleman@ensafe.com</a>

### Notes:

TDEC DSWM = Tennessee Department of Environment and Conservation — Division of Solid Waste Management  
NSA = Naval Support Activity  
USGS = United States Geological Survey  
NAVFAC Midwest = Department of the Navy, Naval Facilities Engineering Command, Midwest

**Comments:** Approximately 1 week prior to the scoping session, Resolution Consultants provided the Base Cleanup Team (BCT) with a draft version of SAP Worksheets #10 (Conceptual Site Model), #11 (Data Quality Objectives) and #17 (preliminary Sampling Design and Rationale), which contained figures showing proposed sampling locations. The October 2012 BCT meeting was held to review the materials and make adjustments per the Team input. The sampling design presented in Worksheet #17 was decided upon and agreed to by the BCT to meet the DQOs and complete the RFI.

### Consensus Decisions:

- (1) Groundwater media would not be sampled in the RFI and may be revisited at a later date, following possible corrective measures when the potential to cross contaminate groundwater with impacted surface soil is lessened.



- (2) An ecological risk assessment will not be conducted, which is consistent with the Activity's RCRA permit requirements. Assessment and remedial decisions will be based on human health risk only.
  
- (3) Surface water media will not be evaluated since there's not a human health exposure pathway and an ecological risk evaluation will not be conducted.

**Action Item:** Revise the draft worksheets #10, #11 and #17 as necessary and re-distribute with remaining SAP worksheets to the Project Team for review and approval.

## **SAP WORKSHEET #10: CONCEPTUAL SITE MODEL**

*(UFP-QAPP Manual Section 2.5.2)*

The Pistol Range (UXO 1) and Trap/Skeet Ranges #1 and #2 (UXO 2) are two of six MRP sites that underwent an SI (Tt, 2010) and have been carried forward for RFI based on the extensiveness of lead and PAH impacts in soil. A brief history of the sites and summary of contaminants detected during the SI are provided below.

### **10.1 Conceptual Site Model for Pistol Range — UXO 1**

#### **10.1.1 Site Location, History, and Physical Features**

The former NSA Mid-South Pistol Range consists of approximately 0.5 acre from the firing line to the backstop soil-berm behind the former target areas and is near the southern border of the installation, east of Singleton Avenue, off the Perimeter Security Patrol Road. The Pistol Range was constructed after 1948 at the former location of a skeet range (known today as the Trap/Skeet Ranges #1 and #2) and was in use until 1994.

Property records indicate that the range, when active, consisted of a berm, shooting stations, targets, and an armory. The firing line maintained 14 firing positions with targets that were operated via a manual, mechanical cable system from behind the firing lines. This included five target lines, one each at 7 yards, 15 yards, 25 yards, and 50 yards. A U-shaped berm, approximately 10 to 15 feet tall and 500 feet long, encompassed the area behind and on both sides of the target lines. Munitions use was limited to small arms ammunition, including 0.22-caliber, 0.30-caliber, 9-mm, 0.38-caliber, and 0.45-caliber ammunition (Malcolm-Pirnie, 2005). Figure 10-1 shows the current conditions for the Pistol Range and Figure 10-2 presents a graphic CSM.

#### **10.1.2 Potential or Known Contamination**

The SI (Tt, 2010) found lead in surface soil on the berm face (0-1 foot below ground surface [bgs]) at concentrations that could present potential risks to human receptors. The exceedances of the project action level (PAL) for lead identified at the Pistol Range were noted in the front face, top, and backside of the berm, and likely are attributed to a combination of fired munitions, contaminant transport via surface water runoff, and the periodic reshaping of the berm. Contributions from activities at the Trap/Skeet Ranges #1 and #2, which surround the Pistol Range berm, may also have contributed to the elevated lead levels in soil. While PAHs were not evaluated during the SI, they are a suspected site contaminant given the significant accumulation of clay pigeons on the berm. Nitroglycerin constituents were not detected during the





**Legend**

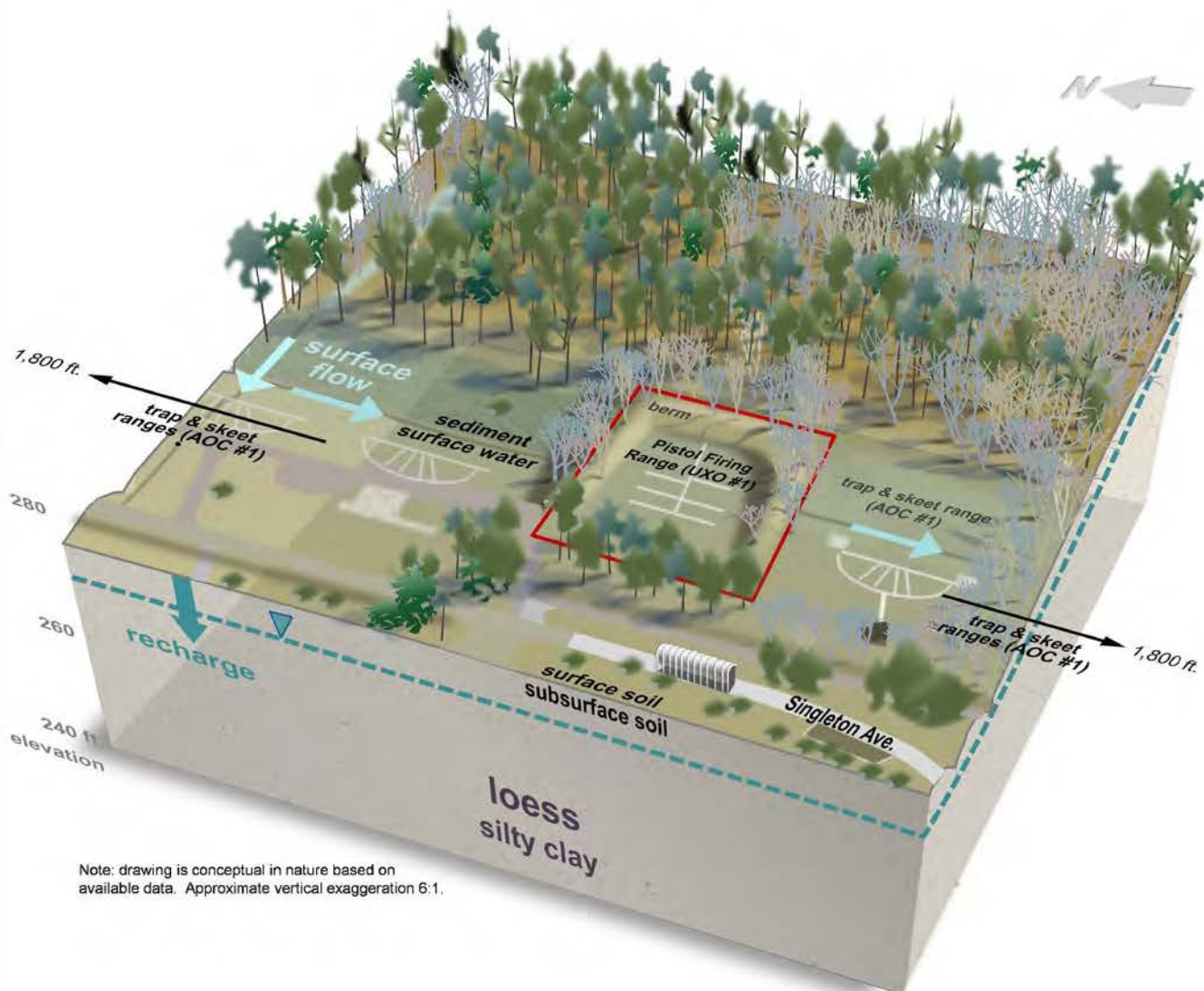
- ⊗ SI Surface Soil Sample for Lead (TT, 2010)
- Road
- Orange outline Pistol Range
- Purple outline Surface Danger Zone
- Blue hatched rectangle Target Lines
- Yellow line Contour (2-ft interval)



Naval Facilities Engineering Command  
RESOLUTION CONSULTANTS

Figure 10-1  
Pistol Range (UXO 1) Site  
Layout  
NSA Mid-South





Note: drawing is conceptual in nature based on available data. Approximate vertical exaggeration 6:1.

### Human Health Exposure Media

**Residential**  
Surface soil  
Sediment

**Site Worker**  
Surface soil

**Construction Worker**  
Surface soil  
Subsurface soil  
Sediment

**Recreation**  
Surface soil



FIGURE 10-2  
CONCEPTUAL SITE MODELS  
AOC #1 AND UXO #1  
NSA MID-SOUTH

REQUESTED BY: L.HUGHES

DATE: 10/22/12

DRAWN BY: BRONSON

DWG NAME: 12676\_B001



SI (Tt, 2010). Expended munitions that did not impact the containment berm may have travelled a much greater distance as indicated by the Surface Danger Zone shown on Figure 10-1. However, given the large areas of undeveloped land that are encompassed by the Surface Danger Zones outside of the current NSA Mid-South property boundaries, the RFI will only focus on impacted soil within the defined boundaries of the Range.

### **10.1.3 Migration Pathways**

As a result of lead in surface soil, a potential risk exists to human health. Lead in surface soil could potentially migrate to subsurface soil and sediment via surface water flow. Clay pigeons may be a potential source of PAHs to soil and sediment via surface water runoff. Lead in surface soil could also potentially infiltrate to the shallow loess water-bearing zone; however, the likelihood was concluded as being low in the SI (Tt, 2010) given the soil's cation-exchange capacity, pH, and total organic carbon. The potential PAH migration from soil to groundwater is also low considering PAHs' low solubility and its affinity for soils. Given this coupled with the potential cross contamination of impacting shallow groundwater while drilling through contaminated soils, the BCT decided not to evaluate groundwater as part of the RFI. Migration pathways are also illustrated in the CSM (Figure 10-2).

### **10.1.4 Receptors, Exposure Pathways, and Future Land Use**

Current human receptors include Navy personnel, Navy-escorted visitors (e.g., contractors conducting environmental or ecological surveys), and trespassers (e.g., authorized personnel who may wander outside of designated areas). The facility's current land uses are expected to remain unchanged in the near future; however, the Base Master Development Plan proposes a recreational reuse of the area that includes the Pistol Range. Therefore, all current potential property reuses, including recreational, will be considered in the risk assessment. In addition, future contractors hired to maintain grounds and perform other maintenance tasks such as sewer repair may be exposed to MC from former range activities.

## **10.2 Conceptual Site Model for Trap/Skeet Ranges #1 and #2 — UXO 2**

### **10.2.1 Site Location, History, and Physical Features**

The former Trap/Skeet Ranges #1 and #2 consist of approximately 79 ac res near the southern border of the installation, east of Singleton Avenue, off the Perimeter Security Patrol Road, and encompassing the former Pistol Range. The Ranges were built around 1943, with Trap/Skeet Range #1 having an unrecorded closure date sometime after World War II and

Trap/Skeet Range #2 closing in 2005. No environmental remedial efforts are documented for closure of the Ranges.

Property records indicate that the trap/skeet Ranges, when active, had a firing line, skeet office and armory, two clay pigeon storage buildings, an ammunition storage building, instruction building, and shooting stations and targets. Munitions use was limited to small arms ammunition, primarily 12- and 20-gauge shotgun shells.

Solid Waste Management Unit (SWMU) 2 is a closed solid waste landfill that served NSA Mid-South and is within the defined boundaries of the former Trap/Skeet Ranges #1 and #2. However, to maintain the integrity of the landfill cap and given its existing institutional controls that restrict access, investigation of the portion of SWMU 2 that lies within the boundaries of the former Trap/Skeet Ranges #1 and #2 was not included in the SI and similarly will be excluded in the RFI. Figure 10-3 shows the current conditions of the former Trap/Skeet Ranges #1 and #2 and Figure 10-2 presents the graphic CSM.

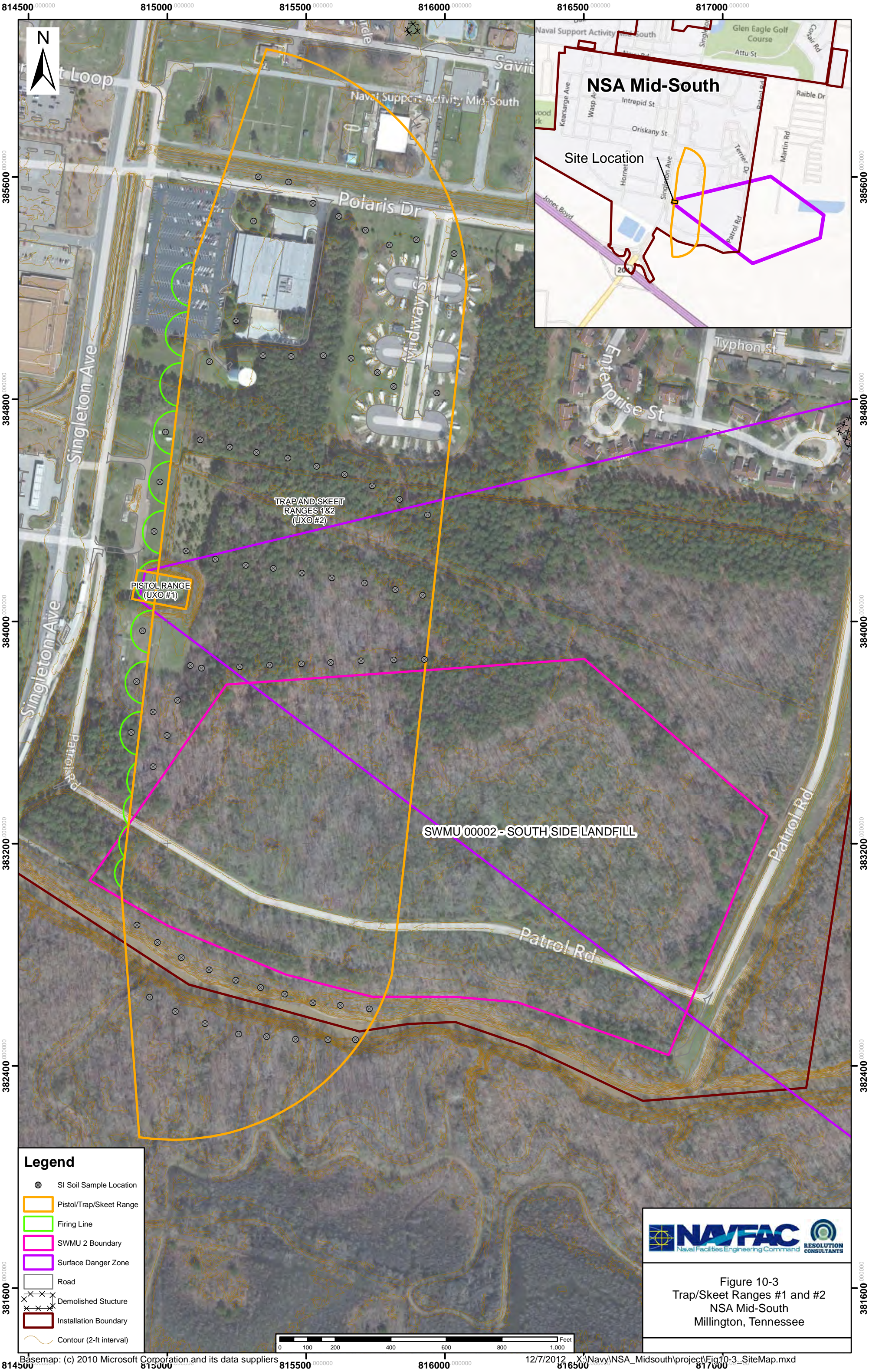
### **10.2.2 Potential or Known Contamination**

The SI (Tt, 2010) identified lead and PAHs primarily within the surface soil (0 to 1 feet bgs) at former Trap/Skeet Ranges #1 and #2, with most exceedances occurring within the central part of the site that extends from the former firing stations to a distance of approximately 600 feet outward in the firing direction. PAH contamination (evaluated as benzo(a)pyrene equivalents) was identified in an area overlapping with the lead-contaminated soil but was limited to a maximum distance of approximately 150 feet from the former firing points. Soil in the northern and southern ends of the site was found not to exceed PALs with the exception of two instances where PAHs were identified at the northern end of the site, which are speculated to be related to parking lot runoff rather than historical site activities.

### **10.2.3 Migration Pathways**

As a result of lead and PAHs in surface soil, a potential risk exists to human health at Trap and Skeet Ranges #1 and #2. The primary migration pathway is considered to be precipitation runoff into nearby drainage ditches. Lead in surface soil could potentially infiltrate to the shallow water-bearing zones in the loess; however, the likelihood is low given the soil's cation exchange capacity, pH, and total organic carbon content. The potential for migration of PAHs from soil to groundwater also is considered low considering the low solubility of PAHs and their affinity for adsorption/absorption to soils. Given this, coupled with the potential cross-contamination of shallow groundwater while drilling through contaminated soils, the BCT decided not to evaluate groundwater as part of the RFI.









#### **10.2.4 Receptors, Exposure Pathways, and Future Land Use**

Current human receptors include Navy personnel that may use the site, Navy-escorted visitors (e.g., contractors conducting environmental or ecological surveys), and trespassers (e.g., authorized personnel who may wander outside of designated areas). The facility's current land uses are expected to remain unchanged in the near future; however, the Base Master Development Plan proposes a recreational reuse of the area that includes Trap/Skeet Ranges #1 and #2. Therefore, all human health exposure scenarios including a future potential recreational reuse scenario will be considered in the risk assessment. In addition, future contractors hired to maintain grounds and perform other maintenance tasks such as sewer repair may be exposed to MC from Range activities.



## **SAP WORKSHEET #11: DATA QUALITY OBJECTIVES/SYSTEMATIC PLANNING PROCESS**

*(UFP-QAPP Manual Section 2.6.1)*

The DQOs presented below were developed in accordance with the *Guidance on Systematic Planning Using the Data Quality Objectives Process* (EPA QA/G-4) (USEPA, 2006) and are intended to fulfill the RFI work plan requirements specified in Attachment 4.2 I of the Facility's RCRA permit.

### **11.1 Problem Statements**

#### *UXO 1 — Pistol Range*

- The SI (Tt, 2010) identified lead concentrations exceeding the human health screening values in surface soil of the Pistol Range berm.
- Soil beneath the Pistol Range berm may be impacted from the former trap and skeet operations that preceded construction of the berm.
- The vertical extent of lead and (or) PAHs within the berm, which are necessary for evaluating appropriate corrective measures, have not been evaluated.
- The human health risk associated with the site are unknown.

#### *UXO 2 — Skeet/Trap Ranges #1 and #2*

- The SI (Tt, 2010) identified lead and PAHs in surface and subsurface soil in concentrations exceeding human health screening values. However, the horizontal and vertical extent of lead and PAHs require further definition for appropriate risk-management decisions.
- Potential impacts to sediment may exist as a result of direct impact from shotgun pellets and (or) skeet fragments, or leaching and subsequent migration from PAH- and (or) lead-impacted soil.
- The human health risk associated with the site is unknown.

### **11.2 Goals of the Study**

The primary goals of the study are to:

1. Collect adequate soil data to determine the horizontal and vertical extents of lead and PAHs at these sites that are in concentrations that exceed the human health benchmarks shown in Worksheet #15.
2. Collect adequate sediment data to determine the extents of lead and PAHs at these sites that are in concentrations that exceed the human health benchmarks shown in Worksheet #15.
3. Delineate the vertical extents of lead and PAHs that are in concentrations that exceed human health benchmarks within the Pistol Range berm so that appropriate soil corrective measures may be evaluated.
4. Gather sufficient data to support a human health risk assessment and determine whether a risk is posed by affected media.

### **11.3 Information Inputs to Resolve the Problem**

Data required for making the decisions include the following:

1. Chemical Data: Soil and sediment will be analyzed for lead and PAHs as prescribed in Worksheet #18. The BCT agreed that lead and PAHs are the contaminants of potential concern (COPCs). Since lead is the primary inorganic munitions constituent, no other metals are being carried forward in the RFI. Nitroglycerin in the form of smokeless powder and ejection of unburned propellants was not identified in soil during the SI (Tt, 2010); therefore, it is not being carried forward as a COPC in the RFI. Worksheet #21 presents the sampling methods to be used and Worksheet #19 presents the analytical methods to be used.
2. Sample Location Data: For spatial presentation of data, all sample points will be surveyed by a Tennessee licensed professional land surveyor for horizontal coordinates and vertical elevations. Horizontal coordinates will be referenced to the North American Datum of 1983 and accurate to 0.1 foot. Depth intervals will be measured by Resolution Consultants field personnel using a tape measure or other device.
3. Miscellaneous laboratory soil parameters: Soil total organic carbon, cation exchange capacity, and pH will be determined for a subset of select samples. These data are

necessary for the evaluation of MC environmental fate and transport if contamination is detected.

4. Field identification/classification of soil types (i.e., lithology and Unified Soil Classification System for grain size, color, plasticity, etc.) Worksheet #21 provides relevant standard operating procedures (SOPs).
5. Screening values: The RFI requires laboratory data from the various media that can be compared to screening values so that appropriate decisions can be made. Laboratory quantitation limits must be low enough to measure lead and PAH concentrations equal to or less than the applicable screening values. The applicable screening values for lead and PAHs are in Worksheet #15.

#### 11.4 Boundaries of the Study

The former Pistol Range (UXO 1) consists of approximately 0.5 acre and contains the former firing lines and a U-shaped soil berm behind the former target areas. UXO 1 is surrounded by the former Trap/Skeet Ranges #1 and #2 (UXO 2), which consist of approximately 79 acres near the southern border of the installation, east of Singleton Avenue, off the Perimeter Security Patrol Road. Visual depictions of UXO 1 and UXO 2 are presented on Figures 10-1 and 10-3, respectively. The horizontal and vertical boundaries as well as the temporal boundaries for the study areas as follows:

***UXO 1 (Pistol Firing Range)*** — The horizontal study boundary encompasses the areas identified on Figures 10-1 and 10-2. The area of the former backstop berm contains surface soil impacts from expended bullets; however, impacts in soil below the uppermost foot of the berm are unknown. Soil deeper than 1 foot below the berm surface and native soil beneath the berm were not evaluated in the SI (Tt, 2010). Given the abundance of lead bullets and accumulated clay pigeons along the berm sides and a history of reworking the berm, the potential for deeper subsurface soil impacts existing with the berm. Given the low mobility of lead and PAHs, significant depth penetration below the berm's base is not expected; therefore, the vertical boundary of UXO 2 is expected to be a maximum of 2 feet below the ground surface of the berm base.

***UXO 2 (Skeet/Trap Ranges #1 and 2)*** — The horizontal study boundary encompasses the areas depicted on Figures 10-2 and 10-3. Areas of interest include those not evaluated in the SI (Tt, 2010), or those "in-between" soil sampling lines that require refinement of the





boundary between impacted and un-impacted areas. The portion of UXO 2 that overlaps with the Southside Landfill (SWMU 2) is not included in the investigation since a land-use control (fencing) for the landfill restricts access.

The SI (Tt, 2010) indicates lead and PAH impacts are predominately in the 0-1 foot bgs soil interval; however, the vertical extent in subsurface soil remains undefined. Given the low mobility of lead and PAHs, significant depth penetration (> 2 feet bgs) is not expected. Therefore, the vertical boundary of UXO 2 is expected to be a maximum of 2 feet bgs.

Sediment within the drainage ditches that cross the site is a medium of interest that may be contaminated resulting from the receipt of shotgun pellets and/or skeet fragments during range operations or subsequent migration of nearby surface soil contaminants.

The temporal boundaries of lead and PAHs are anticipated to remain relatively unchanged (stable) over the course of time needed to conduct the environmental investigations and into the foreseeable future; therefore, no temporal constraints exist.

### **11.5 Analytical Approach**

The approach described below explains the decision rules that the BCT agreed upon for the data collection proposed at UXO 2 and UXO 1. The decision rules are intended to define the nature and extent of lead and PAHs in soil and sediment and will be based on the following:

1. The need for additional delineation of the COPCs will be determined using PALs, which are the discriminators between contaminated and uncontaminated media for human health risk evaluations. The Facility's RCRA permit (Attachment 4.5;I) specifies that, screening levels shall be concentration levels that satisfy the following criteria:
  - a) Are derived in a manner consistent with the U.S. Environmental Protection Agency (USEPA) guidelines for assessing human and environmental health risks from hazardous constituents;
  - b) Are based on scientifically valid studies conducted in accordance with the Toxic Substances Control Act (TSCA) Good Laboratory Practice Standards, or equivalent;

- c) For human health screening levels to address carcinogens, represents a concentration associated with an excess upper bound lifetime cancer risk of  $1 \times 10^{-6}$  for carcinogens due to continuous constant lifetime exposure; and
- d) For human health screening levels to address systemic toxicants, represents a concentration to which the human population (including sensitive subgroups) could be exposed on a daily basis that is likely to be without appreciable risk of deleterious effects during a lifetime.

The screening levels that will be used for soil and sediment PALs are the USEPA Regional Screening Levels from:

([http://www.epa.gov/reg3hwmd/risk/human/rb-concentration\\_table/index.htm](http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm)).

Analytical methods were chosen that would achieve the best detection limits to answer the study questions. Good-faith efforts were undertaken to ensure that contaminants can be measured at the lowest achievable levels for the applied method. However, in some cases, current technology used by the analytical methods cannot achieve all of the PALs. When PALs are less than laboratory's detection limit, all non-detected results will be reported to detection limit and any limitations on data use that result from having detection limits greater than PALs will be described in the project report. The project report will describe any uncertainties that result from having detection limits greater than the PALs. Particular scrutiny will be applied to these situations when determining whether project objectives have been obtained.

Pre-established background concentrations for lead and PAHs in soil will also be considered during the data evaluation. Step-out sampling will be considered as a means to determine contamination extent; an approach will be proposed, discussed, and agreed upon before scoping and planning details pertaining to any future supplemental sampling efforts. In cases where analytical methods cannot achieve all of the PALs, any non-detect values that are above the PALs will not be considered for additional step-out sampling. The PALs, analytical methods, and target analytes are listed in Worksheet #15

2. If lead and PAH concentrations are less than PALs listed in Worksheet #15, then the extent of contamination will be considered fully defined.

3. If the BCT determines, based on a combination of the SI and proposed RFI data, that the presence of PAHs and lead have been adequately delineated in all environmental media, then assessment activities will be considered complete and the human health risk evaluation will be conducted and the RFI report will be initiated. At a minimum, this evaluation must consider the following factors relative to the existing CSM for each environmental medium:
  - Frequency of detection of each constituent
  - Frequency and magnitude of screening criteria exceedances for each constituent
  - Background concentrations of metals that exceed screening criteria
  - Magnitude of concentrations within, and on the perimeter of, the investigated areas
  - Identities of COPCs and their estimated contributions to unacceptable levels of risk
  
6. Detections that exceed their respective PALs will be screened against existing background values for lead and PAHs in soil (E/A&H, 1996). If lead or PAH concentrations exceed their PALs for soil but are below the background screening value, then these media will be considered unaffected by former site operations.
  
7. If concentrations of lead or PAHs in sediment exceed a PAL, then a background evaluation of the media will be conducted to determine whether contaminants are site-related.

## 11.6 Human Health Risk Assessment

The human health risk assessment will evaluate the following media, receptors, and exposure pathways:

Media		Receptors				
		Exposure Route	Residents	Commercial Workers	Construction Workers	Recreational Users
Surface Soil		Ingestion	X			X
		Inhalation	X			X
		Dermal contact	X			X
Subsurface Soil		Ingestion			X	
		Inhalation			X	
		Dermal contact			X	
Sediment		Ingestion	X			
		Inhalation				
		Dermal contact	X	X	X	

If risks estimated using representative exposure point concentrations are unacceptable from soil or sediment (i.e., hazard quotients are greater than 1, and/or incremental lifetime cancer risks are greater than  $1 \times 10^{-4}$ ), the Navy will proceed to a corrective measures study to evaluate remedial alternatives for the site; otherwise no further investigation or action with respect to chemical contamination in the appropriate media will be recommended.

### **11.7 Performance Criteria**

The objective of this section is to complete the following:

- Identify potential sources of study error (i.e., field error, analytical error)
- Establish and identify the methods used to reduce potential sources of error
- Determine how decision errors will be managed during the project

#### *Sampling Strategy*

The sampling strategy is designed to characterize the nature and extent of contamination and collect adequate data to conduct a human health risk evaluation.

#### *Sources of Error*

Sources of error may be divided into two main categories: sampling errors and measurement errors. A sampling error occurs when the sampling design, planning, and implementation do not provide for a representative range of heterogeneity at the site. A measurement error occurs because of performance variance from laboratory instrumentation, analytical methods, and operator error. The USEPA identifies the combination of all these errors as a "total study error" (USEPA, 2006). One objective of the investigation is to reduce the total study error so that decision-makers can be confident that the data collected accurately represent the chemical characteristics of the site.

#### *Managing Decision Error*

The investigation will utilize decision-error minimization techniques in sampling design, sampling methodologies, and laboratory measurement of COPCs. Possible decision errors will be minimized during the field investigation by using the following methods:

- Use of standard field sampling methodologies (as discussed in Worksheets #18 and #21).



- Use of applicable analytical methods (discussed in Worksheets #23, #24, and #25) for sample analysis by a competent analytical laboratory certified by the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) to reduce measurement errors.
- Confirm analytical data to identify and control potential laboratory error and sampling error by using spikes, blanks, and replicated samples.

Decision errors are based on sample design and measurement errors. Assuming that the best possible professional judgment was used to develop the sampling plan (i.e., position sampling locations), the most significant decision errors will be associated with field and laboratory techniques involved in the collection and analysis of the data.

#### *Sampling Methodologies and Procedures*

Possible decision errors generated by sampling errors will be minimized during the field investigation by applying standardized field sampling methodologies (discussed in Worksheets #18, #20, #21, and #22). Sampling activities will be performed in accordance with the SOPs specified in this SAP.

#### *Managing Laboratory Sampling Error*

Control of potential laboratory measurement error and sampling error will be minimized using spikes, blanks, and duplicates. Sampling error may be introduced when the laboratory chemist selects a single portion of the field sample for laboratory analysis. However, this issue is less relevant to the discrete sampling approach as sub-sampling is generally not implemented at the laboratory.

The laboratory will provide full electronic data deliverable files, portable document format (PDF) files of the data deliverables for all project data, and a hard copy of data deliverables for all results including results from secondary subcontract laboratories. Designated samples will be used to obtain necessary sub-samples for laboratory quality control (QC) measurements (i.e., analytical sample duplicate and sample matrix spike/matrix spike duplicate [MS/MSD]). Tasks will be completed using the laboratory SOPs.



Data quality will be evaluated as part of the verification/validation and the data usability processes described in Worksheets #34 through #36. Failure to meet validation targets or limitations on data use identified during a data usability assessment shall be described in the project report. PDF copies of all analytical data packages will be stored on CD-ROM, archived in the NAVFAC Midwest Administrative Record, and uploaded onto the Naval Installation Restoration Information Solution (NIRIS) system at the close of the project. All other data generated in the field and reports generated for the project will be stored as computer readable data files by Resolution Consultants.

### **11.8 Sampling Design**

The sampling design for the site has been developed to optimize resources and generate data to satisfy the DQOs. The sampling design, rationale, and locations are summarized in Worksheets #17 and #18. These worksheets identify where soil and sediment samples will be collected and the analyses to be conducted for each sample.



## SAP WORKSHEET #12: FIELD QUALITY CONTROL SAMPLES

(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table — Field Quality Control Samples				
Quality Control Sample	Analytical Group	Frequency	Data Quality Indicators	Measurement Performance Criteria
Equipment Rinsate Blanks	PAHs and Lead	One per 20 field samples per matrix per sampling equipment <sup>1</sup>	Accuracy/Bias/Contamination	No analytes > ½ LOQ, except common lab contaminants, which must be < LOQ
Field Duplicates	PAHs and Lead	One per 20 field samples	Precision	Values > 5X LOQ: RPD must be ≤30 (aqueous) <sup>2</sup> ; ≤50 (solids) <sup>2,3</sup> .
Matrix Spike/Matrix Spike Duplicate	PAHs and Lead	One pair per 20 field samples	Accuracy/Bias/Precision	Percent recoveries — DoD QSM Limits RPD must be ≤ 30 (PAHs) RPD must be ≤ 20 (lead)
Cooler Temperature Indicator	All	One per cooler	Representativeness	Temperature less than 6 degrees Celsius

### Notes:

<sup>1</sup> Equipment rinsate blanks will be collected if decontamination is required and will not apply if dedicated equipment is used.

<sup>2</sup> If duplicate values for PAHs are less than five times the LOQ, the absolute difference should be less than or equal to two times the LOQ.

<sup>3</sup> If duplicate values for lead is less than five times the LOQ, the absolute difference should be less than or equal to four times the LOQ.

PAHs = Polynuclear aromatic hydrocarbons

LOQ = Limit of Quantitation

RPD = Relative Percent Difference

DoD QSM = *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 4.2, October 2010 or the most recent version at the time of sampling.





## SAP WORKSHEET #13: SECONDARY DATA CRITERIA AND LIMITATIONS TABLE

(UFP-QAPP Manual Section 2.7)

Secondary Data Criteria and Limitations Table				
Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
Historical Background Information, Data	<i>Site Inspection Report for MRP Site Inspections at NAVFAC Midwest and BRAC Program Management Office Southeast Munitions Response Sites and Areas of Concern; Naval Support Activity Mid-South; Millington, Tennessee, September 2010</i>	<i>Originating Organization: Tetra Tech NUS Data Types: Background information Data Collection Dates: 2010</i>	Background information was used in planning of the sampling effort	None
Historical Background Information, Data	<i>Sampling and Analysis Plan for Field Sampling Plan and Quality Assurance Project Plans for Small Range Site Inspection; Millington Naval Support Activity, December 1, 2009</i>	<i>Originating Organization: Tetra Tech NUS Data Types: Background information Data Collection Dates: 2009</i>	Background information from the Preliminary Assessment	None

## **SAP WORKSHEET #14: FIELD PROJECT TASKS**

*(UFP-QAPP Manual Section 2.8.1)*

Project-specific SOPs and Field Forms for field tasks referenced in this worksheet are identified by title in Worksheet #21 and copies of each SOP are in Appendix A. All investigative activities will be recorded in a field log book(s) in accordance with SOP3-02. The field tasks are as follows:

- Site Preparation
  - Mobilization/Demobilization
  - Utility Clearance
  - MEC avoidance
- Field Investigation
  - Soil Sampling
  - Sediment Sampling
  - Land Surveying
  - Waste Handling
  - Decontamination
  - Field Documentation
- Laboratory Analysis
- Data Validation and Management
- Data Evaluation
- Reporting

### **FIELD TASKS**

**Mobilization/Demobilization** — Mobilization will consist of the delivery, assembly (in satisfactory working order), and secure storage of necessary equipment, materials, and supplies, along with the acquisition of personnel and vehicle base access badges. The Resolution Consultants Field Team Leader (FTL) or designee will coordinate with the NSA Mid-South point of contact (POC) to identify appropriate locations for the temporary storage of equipment and supplies.

Demobilization will consist of the prompt and timely removal of equipment, materials, and supplies from the site at the completion of fieldwork. Demobilization also includes the cleanup and removal of waste generated during the investigation.

**Utility Clearance** — A minimum of 2 weeks prior to the commencement of any intrusive activities, Resolution Consultants will coordinate utility clearance with the NSA Mid-South POC and



Tennessee One Call, in accordance with SOP-3-01. Utilities will be marked at proposed sampling locations before intrusive sampling is initiated. The Resolution Consultants FTL will document the utility clearance process and obtain all required approvals, as deemed necessary by NSA Mid-South. Utilities that are identified in the field, but not shown or incorrectly shown on the work approval documentation, will be marked directly on the document and returned to the NSA Mid-South POC for inclusion in the Geographic Information System database.

### **Munitions and Explosives of Concern Avoidance**

Although the likelihood of encountering MEC or material potentially presenting an explosive hazard is low, a UXO-qualified technician will be onsite during intrusive activities performed at UXO 1 as a conservative measure to provide MEC avoidance support. MEC avoidance services are only required at UXO 1 based on the *Memorandum: Explosive Safety Submission Determination Request for Trap and Skeet Range (UXO 2) and Pistol Range (UXO 1) at Naval Support Activity Mid-South, Millington, Tennessee* (DoD, 2012).

MEC avoidance services will be performed by a DoD Explosive Safety Board TP-18 qualified UXO Technician II or equivalent using a handheld analog detector. If potential MEC is encountered, personnel will evacuate the area, the location and description of the object will be recorded, the responsible Explosives Safety officer, the Navy RPM, NSA Mid-South POC, and Naval Ordnance Safety and Security Activity will be notified, and removal of the object(s) will be coordinated with Explosive Ordnance Disposal detachment.

**Soil Sampling** — The sampling and analysis program is outlined in Worksheets #17 and #18. Surface soil sampling will be collected from 0 to 12 inches bgs and subsurface soil sampling will be collected at 1-foot intervals thereafter. Sample collection and handling will be in accordance with SOP-3-03A, SOP-3-04A, and SOP-3-21 and as described in Worksheet #21. A soil-boring log will be prepared for monitoring well borings and those that penetrate the top of the berm. Boring logs will contain soil descriptions and all relevant information, observations, depth to water, etc. Sample depths will be included on each log. After sampling, hand-auger boreholes will be backfilled to grade using soil cuttings removed from the borehole and/or bentonite grout.

**Sediment Sampling** — A sample log will be prepared for each location with descriptions and all relevant information and observations. Sample depths will be included on each log. After sampling, each hole will be backfilled as needed using sediment removed from the hole.

**Land Surveying** — All soil and sediment sample locations will be surveyed by a professional surveyor licensed in the State of Tennessee. Wooden stakes with the sample station ID will be placed at each sample location at the conclusion of sampling. The Resolution Consultants FTL or designee will provide the surveyor with the coordinate designation nomenclature and all coordinate systems will be pre-approved prior to commencement of the survey. All measurements will be reported and recorded in U.S. Survey Feet. The surveyor will find and use existing survey controls. It is anticipated that adequate control is located within 1 mile of the site. Horizontal position should be referenced to Tennessee State Plane Coordinates and vertical elevation will be referenced to North American Vertical Datum of 1983 and accurate to 0.01 foot. The surveyor will document all work in a clear, legible, and complete manner. The field record will contain a complete description of the nature and location of the new and existing points. The record will also include a sketch of the point locations and the benchmark witness points for both project control and local control.

**IDW Management** — Investigative derived waste (IDW) management and sampling activities will be conducted to properly stage IDW and characterize it for disposal. IDW generated from field investigation activities will include soil cuttings, decontamination fluids, and potentially construction debris (e.g., concrete and underground pipes). During the field investigation, waste minimization practices will be implemented to the extent practicable. Soil excavated from trenches during the UXO 1 berm assessment will be placed back into the trench from which it originated; therefore, trench soil will not be considered IDW. IDW will be managed onsite in U.S. Department of Transportation-approved 55-gallon steel drums. Wastes such as consumable materials, supplies, and equipment will be disposed of as municipal trash in an offsite dumpster.

IDW will be characterized for disposal by evaluating environmental sampling data and by collecting and analyzing waste characterization samples. If environmental sampling data are insufficient to characterize IDW for disposal, then waste characterization samples will be collected to characterize soil and decontamination fluids for disposal. IDW characterization data will be removed and transported to an appropriate disposal facility within 90 calendar days of completing the field investigation. IDW will be managed in accordance with the procedures outlined in SOP 3-05.

Personal protective equipment, including gloves, wipes, discarded paper towels, and disposable equipment (i.e., tubing), will be bagged and properly discarded in an appropriate on-base solid waste Dumpster.

## **Field Decontamination**

Equipment decontamination activities will be conducted on reusable equipment that comes in contact with site soil or sediment to prevent cross-contamination of sampling equipment. Decontamination fluids will be collected in 55-gallon drums and managed in accordance with SOP 3-06.

Steam-washing is the preferred method for decontaminating non-sampling equipment. Backhoe buckets, direct-push rods, and augers will be placed within secondary containment and steam-washed to remove residual soil and chemicals from the equipment surface.

Reusable sampling equipment will be decontaminated using a five -step decontamination process. Reusable equipment includes drill hole saw bits, trowels, and split-spoon samplers. Five buckets will be placed in secondary containment and in the following series: 1) detergent wash using alconox/liquinox, 2) water bath, 3) isopropyl alcohol (70% by volume) rinse, 4) water bath, and 5) water rinse. Reusable sampling equipment will be cleaned of residue by washing the equipment surface beginning at Step 1 and proceeding to Step 5.

## **Analytical Tasks**

Gulf Coast Analytical Laboratories, a DoD ELAP-accredited laboratory, will perform chemical analysis. Copies of pertinent laboratory accreditations are in Appendix C. Analyses will be performed in accordance with the analytical methods identified in Worksheets #23 and #30. The subcontract laboratory will perform the chemical analyses per laboratory-specific SOPs identified in Worksheet #23 and in Appendix D.

The subcontracted laboratory will provide fully validatable data packages, which will include summary forms containing all method-specific quality control information and raw data. Sediment results will be reported by the laboratory on an adjusted dry-weight basis, as applicable to the testing method. Results of percent moisture will be reported in each analytical data package and associated electronic data-deliverable files. This information also will be captured in the project database, which will eventually be uploaded to the NIRIS database.

## **Data Management**

- **Data Handling and Management** — Upon completion of field activities, all secondary data from field sampling forms will be organized by date and entered into Excel files for electronic archiving/data evaluation. The field forms, chain of custody, air bills, and

logbooks will be placed in the project files after the completion of the field program. The field logbooks for this project will be used only for this site, and also will be categorized and maintained in the project files after the completion of the field program. After data are validated, the electronic data results will be uploaded into the Resolution Consultants database for use in data evaluation and subsequent report preparation. The project database will be located on a secure network, which is password protected. Final validated environmental data and special data will be uploaded into the NIRIS database.

- **Data Tracking and Control** — The Task Order Manager (TOM) (or designee) is responsible for the overall tracking and control of data generated for the project. Data are tracked from their generation to their archiving in the project specific files. The project chemist (or designee) is responsible for tracking the samples collected and shipped to the contracted laboratory. Upon receipt of the data packages from the analytical laboratory, the project chemist will oversee the data validation effort, which includes verifying that the data packages are complete and that results for all samples have been delivered by the analytical laboratory.

Resolution Consultants shall submit all Administrative Record Files, Site Files, and Post Decision Files in accordance with the specifications defined in the NAVFAC Environmental Restoration Program Recordkeeping Manual (Naval Facilities Engineering Command, September 2009). Additionally, Resolution Consultants will update and manage the project related documents, data, and maps in NIRIS. Project related spatial data including maps, models, and associated collected or created data will also be uploaded into NIRIS. All documentation submittals for NIRIS will be coordinated with the RPM.

- **Data Storage, Archiving, and Retrieval** — After the data are validated, the data packages are entered into the Resolution Consultants Navy CLEAN file system and archived in secure files. The field records, including field log books, sample logs, chain-of-custody records, and field calibration logs, will be submitted by the Resolution Consultants FTL to be entered into the Navy CLEAN file system before archiving in secure project files. Project files are audited for accuracy and completeness. Project files will be kept in a secured, limited access area and at the completion of the Navy contract, the records will be



stored by Resolution Consultants and eventually transferred to the Navy. Final validated environmental data, special data, will be uploaded into the NIRIS database.

### **Documentation and Reporting**

- All field activities associated with UXO 2 and UXO 1 will be incorporated into the RFI report, which will include a summary of the completed tasks, well-construction logs, field records, and deviations that may have occurred from this SAP.
- All UXO 2 and UXO 1 data will be compiled and evaluated in the RFI report and submitted to the BCT members for review, comment, and finalization. The human health risk assessment will be submitted either as a standalone document or included with the corrective measures study.
- All administrative record files, post decision files, and site files will be submitted to the NIRIS data management system in accordance with the *Environmental Restoration Recordkeeping Program Manual, Appendix G, NAVFAC Contractor Work Instruction* (NFESC, September 2009).



## SAP WORKSHEET #15: REFERENCE LIMITS AND EVALUATION TABLE

(UFP-QAPP Manual Section 2.8.1)

Matrix: Soil and Sediment Analytical Group: Low-Level PAHs via SIM and Lead							
Analyte	CAS No	Project Action Level (mg/kg)	Project Action Level Reference (mg/kg)	Project Quantitation Limit Goal (mg/kg)	Laboratory Limit of Quantitation <sup>1</sup> (mg/kg)	Laboratory Limit of Detection <sup>1</sup> (mg/kg)	Laboratory Detection Limit <sup>1</sup> (mg/kg)
2-Methylnaphthalene	91-57-6	230	RSL	77	3.3	1.65	0.99
Acenaphthene	83-32-9	3,400	RSL	1,133	3.3	1.65	0.83
Acenaphthylene	208-96-8	3,400 <sup>2</sup>	RSL	1,133	3.3	1.65	0.95
Anthracene	120-12-7	17,000	RSL	5,667	3.3	1.65	0.51
<b>Benzo(a)anthracene</b>	<b>56-55-3</b>	<b>0.15</b>	<b>RSL</b>	<b>0.05</b>	<b>3.3</b>	<b>1.65</b>	<b>0.59</b>
<b>Benzo(a)pyrene</b>	<b>50-32-8</b>	<b>0.015</b>	<b>RSL</b>	<b>0.005</b>	<b>3.3</b>	<b>1.65</b>	<b>0.66</b>
<b>Benzo(b)fluoranthene</b>	<b>205-99-2</b>	<b>0.15</b>	<b>RSL</b>	<b>0.05</b>	<b>3.3</b>	<b>1.65</b>	<b>0.5</b>
Benzo(g,h,i)perylene	191-24-2	1,700 <sup>3</sup>	RSL	567	3.3	1.65	0.65
Benzo(k)fluoranthene	207-08-9	1.5	RSL	0.5	3.3	1.65	0.48
Chrysene	218-01-9	15	RSL	5	3.3	1.65	0.45
<b>Dibenzo(a,h)anthracene</b>	<b>53-70-3</b>	<b>0.015</b>	<b>RSL</b>	<b>0.005</b>	<b>3.3</b>	<b>1.65</b>	<b>0.51</b>
Fluoranthene	206-44-0	2,300	RSL	770	3.3	1.65	0.42
Fluorene	86-73-7	2,300	RSL	770	3.3	1.65	0.57
<b>Indeno(1,2,3-cd)pyrene</b>	<b>193-39-5</b>	<b>0.15</b>	<b>RSL</b>	<b>0.05</b>	<b>3.3</b>	<b>1.65</b>	<b>1</b>
<b>Naphthalene</b>	<b>91-20-3</b>	<b>3.6</b>	<b>RSL</b>	<b>1.2</b>	<b>3.3</b>	<b>1.65</b>	<b>0.97</b>
Phenanthrene	85-01-8	1,700 <sup>3</sup>	RSL	567	3.3	1.65	0.72
Pyrene	129-00-0	1,700	RSL	567	3.3	1.65	0.5
Lead	7439-92-1	400	RSL	133	0.05	0.04	0.03

### Notes:

<sup>1</sup> Laboratory quantitation, detection, and method detection limits are provided by Gulf Coast Analytical Laboratories and may vary during the course of the investigation.

<sup>2</sup> Surrogate value from Acenaphthene

<sup>3</sup> Surrogate value from Pyrene

PAHs = Polynuclear aromatic hydrocarbons

SIM = Selective ion monitoring

mg/kg = Milligrams per kilogram

RSL = USEPA Regional Screening Levels (May 2012); Region 9; <http://www.epa.gov/region9/superfund/prg/rsl-table.html>

**Bold cells** indicate that one or both Project Action Levels (PALs) are between the laboratory limit of quantitation and detection limit.

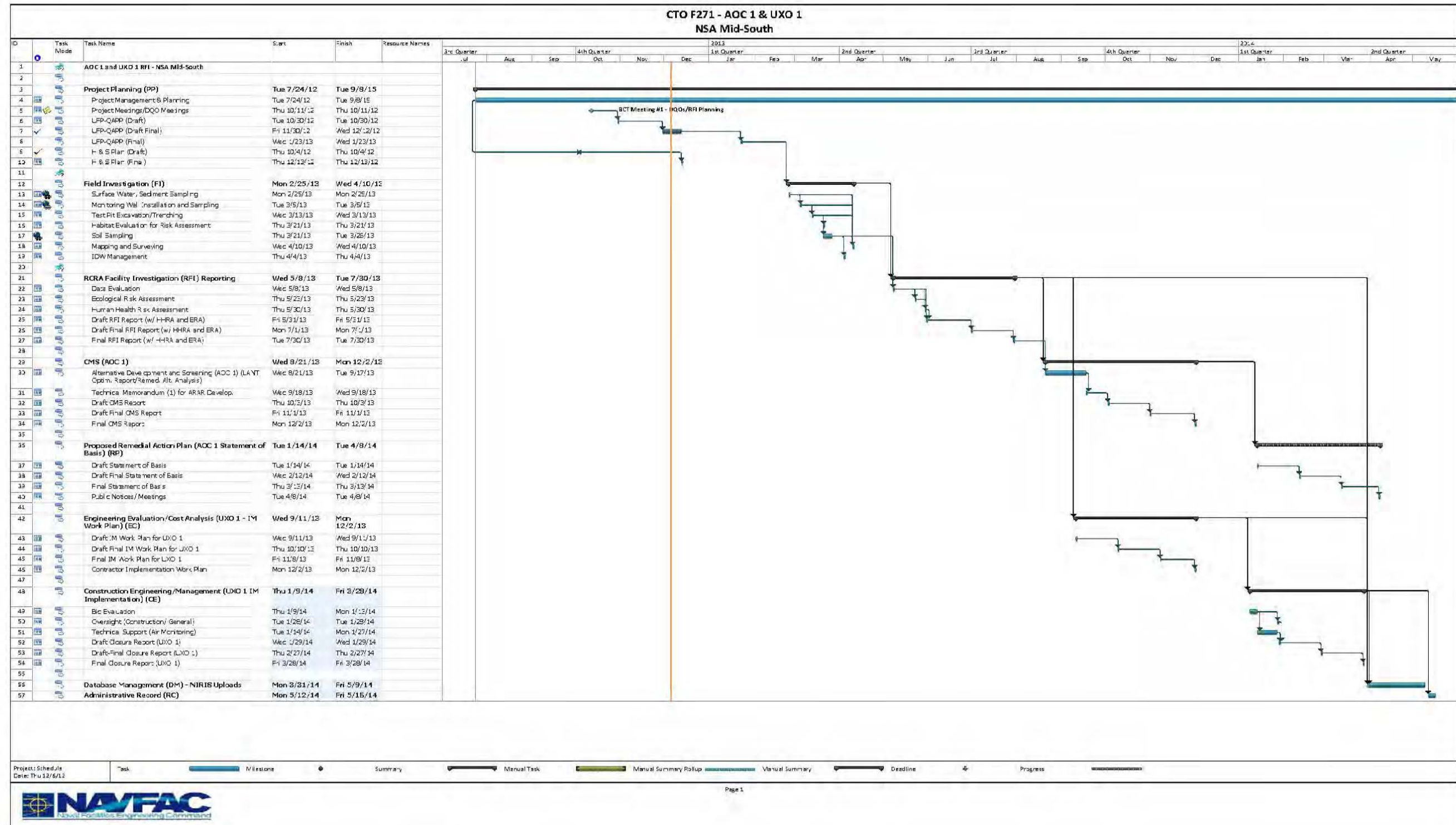
**Bold and shaded cells** indicate that one or both PALs are less than the detection limit. All results will be reported to detection limits and any limitations on data use that result from having detection limits greater than PALs will be described in the project report. The Partnering Team will describe in the project report any uncertainties that result from having detection limits greater than the PALs. Particular scrutiny will be applied to these situations when determining whether project objectives have been obtained.





SAP WORKSHEET #16: PROJECT SCHEDULE/TIMELINE TABLE

(UFP-QAPP Manual Section 2.8.2)





## **SAP WORKSHEET #17: SAMPLING DESIGN AND RATIONALE**

*(UFP-QAPP Manual Section 3.1.1)*

### **17.1 General Sampling Design and Rationale**

The objectives of the sampling rationale described below are (1) to collect adequate soil and sediment data for characterizing the nature and extent of any PAH or lead impacts, and (2) to assess the data against human health risk benchmarks. The sampling design and rationale was agreed to by the BCT during the October 2012 meeting as necessary to meet the above objectives and the DQOs discussed in Worksheet 11. Although MEC are not expected to be encountered, a UXO-qualified technician will be onsite during intrusive activities performed at UXO 1 as a conservative measure to provide anomaly avoidance support to the field team in accordance with the Memorandum: *Explosive Safety Submission Determination Request for Trap and Skeet Range (UXO 2) and Pistol Range (UXO 1) at Naval Support Activity Mid-South, Millington, Tennessee (DoD, 2012)*. The sampling design for the sites is the following:

#### ***UXO 2 — Trap/Skeet Ranges #1 and #2***

- Assess the horizontal and vertical extent of PAH and lead contamination in soil and sediment at the former trap/skeet ranges.

#### ***UXO 1 — Pistol Firing Range***

- Assess the horizontal and vertical extent of lead and PAH impacts in soil within, adjacent to, and beneath the backstop berm.

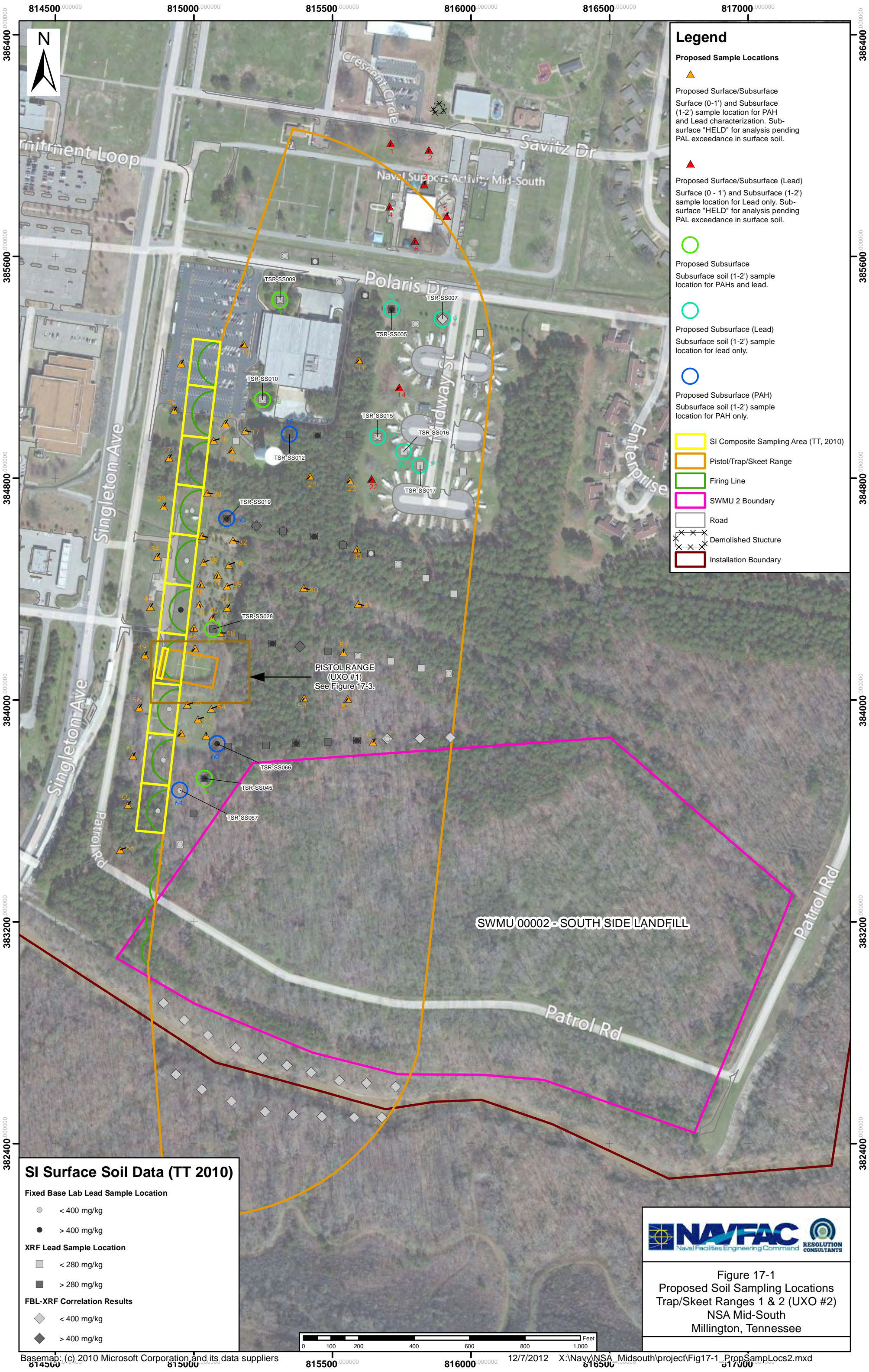
PAHs and lead were established as COPCs in the SI (Tt, 2010) and adopted by the BCT. Analytical data will be compared to the human health benchmarks listed in Worksheet #15 to determine if horizontal and vertical step-out sampling will be necessary to establish the nature and extent of contamination.

### **17.2 Site-Specific Sampling Design**

#### **Analytical Testing**

The proposed RFI field data-collection program is presented on Figures 17-1 through 17-3 and discussed for each media below. The information developed in previous sections of this worksheet and presented in the SI (Tt, 2010) was used to develop the soil-sampling program along with input from the BCT during the October 2012 meeting.





Legend

Proposed Sample Locations

▲  
Proposed Surface/Subsurface  
Surface (0-1') and Subsurface  
(1-2') sample location for PAH  
and Lead characterization. Sub-  
surface "HELD" for analysis pending  
PAL exceedance in surface soil.

▲  
Proposed Surface/Subsurface (Lead)  
Surface (0 - 1') and Subsurface (1-2')  
sample location for Lead only. Sub-  
surface "HELD" for analysis pending  
PAL exceedance in surface soil.

○  
Proposed Subsurface  
Subsurface soil (1-2') sample  
location for PAHs and lead.

○  
Proposed Subsurface (Lead)  
Subsurface soil (1-2') sample  
location for lead only.

○  
Proposed Subsurface (PAH)  
Subsurface soil (1-2') sample  
location for PAHs only.

SI Composite Sampling Area (TT, 2010)

Pistol/Trap/Skeet Range

Firing Line

SWMU 2 Boundary

Road

Demolished Structure

Installation Boundary

SI Surface Soil Data (TT 2010)

Fixed Base Lab Lead Sample Location

- < 400 mg/kg
- > 400 mg/kg

XRF Lead Sample Location

- < 280 mg/kg
- > 280 mg/kg

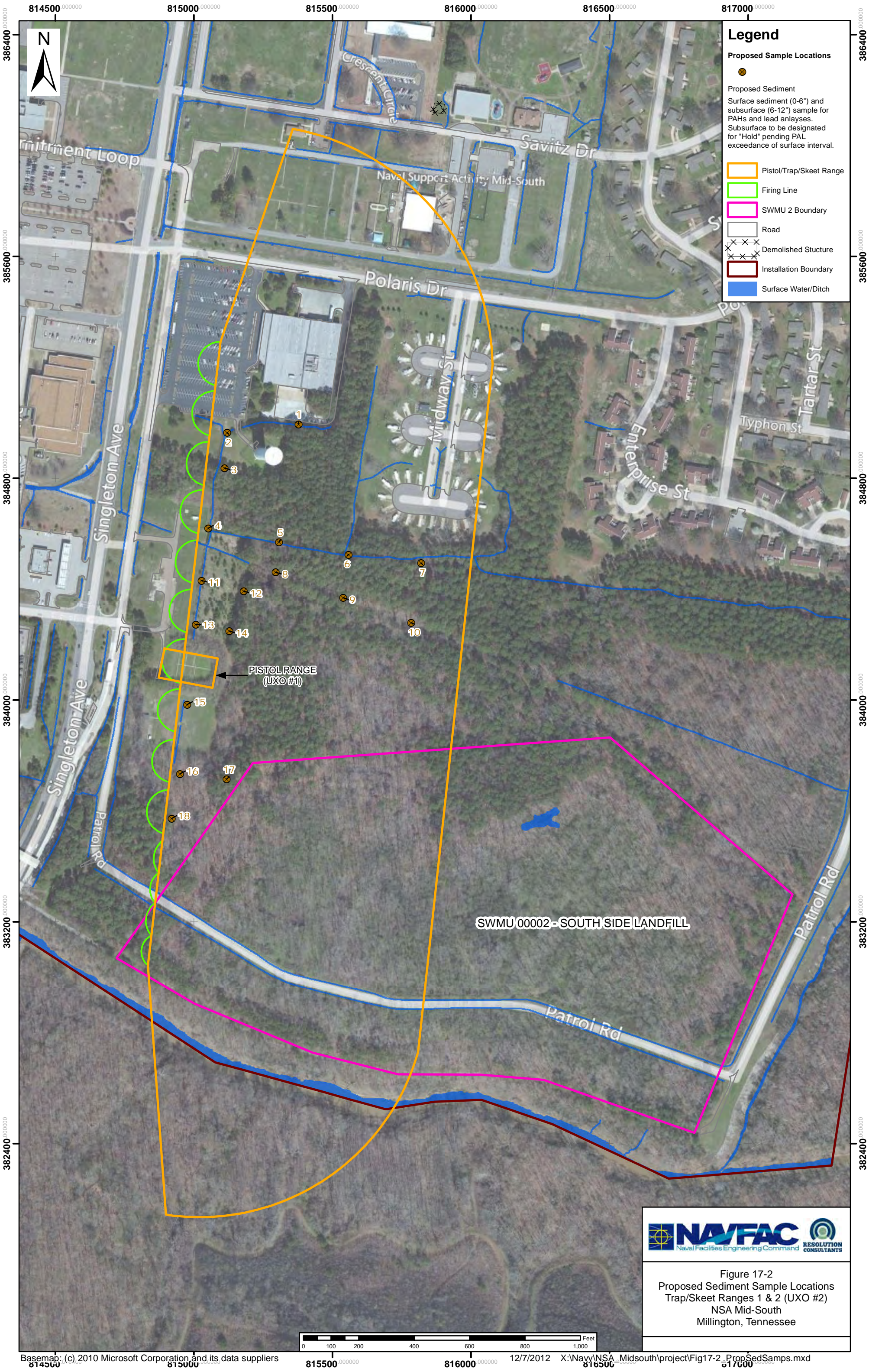
FBL-XRF Correlation Results

- ◆ < 400 mg/kg
- ◆ > 400 mg/kg



Figure 17-1  
Proposed Soil Sampling Locations  
Trap/Skeet Ranges 1 & 2 (UXO #2)  
NSA Mid-South  
Millington, Tennessee





Legend

Proposed Sample Locations

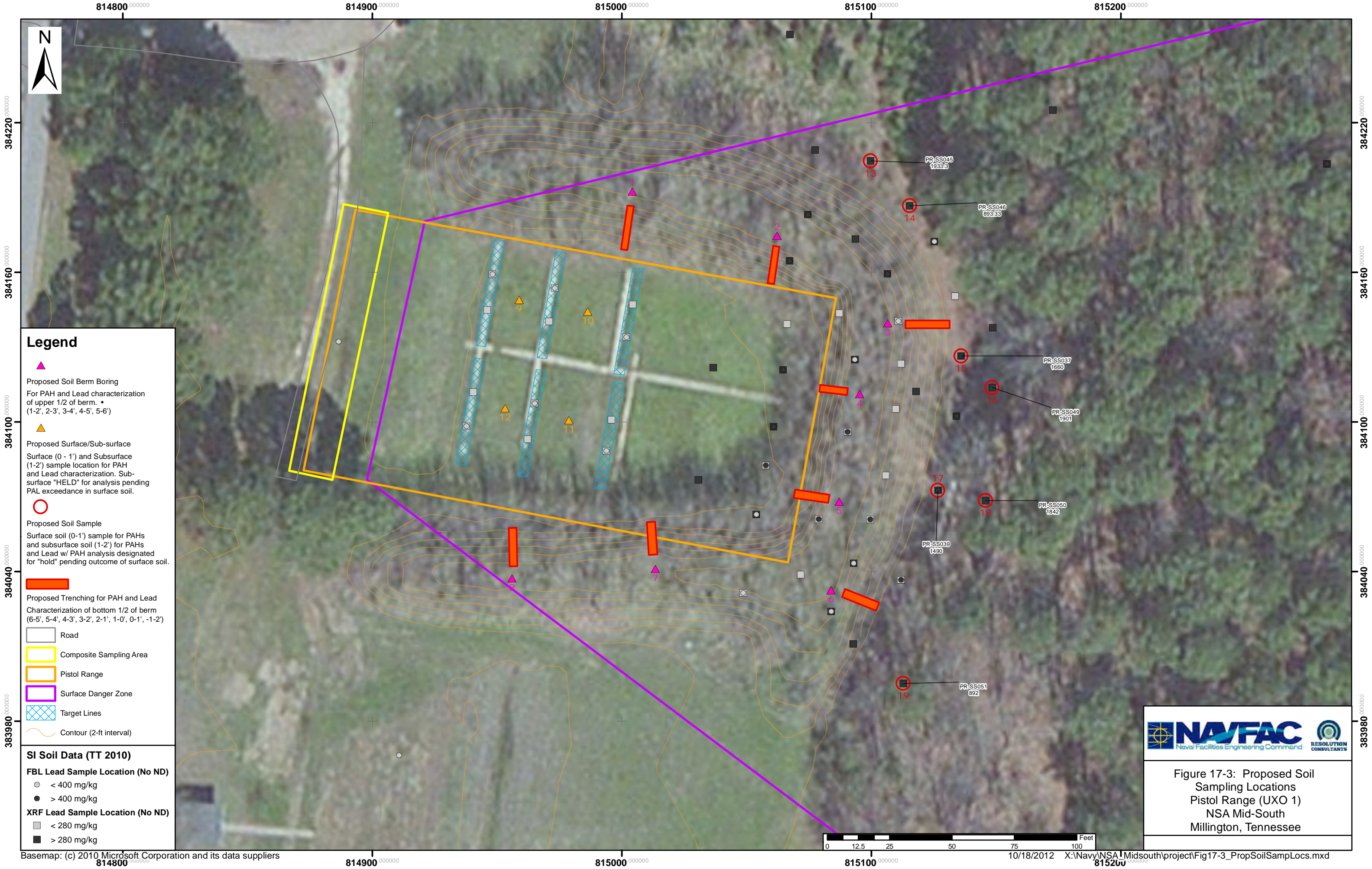
Proposed Sediment  
Surface sediment (0-6") and  
subsurface (6-12") sample for  
PAHs and lead analyses.  
Subsurface to be designated for  
"Hold" pending PAL  
exceedance of surface interval.

- Pistol/Trap/Skeet Range
- Firing Line
- SWMU 2 Boundary
- Road
- Demolished Structure
- Installation Boundary
- Surface Water/Ditch



Figure 17-2  
Proposed Sediment Sample Locations  
Trap/Skeet Ranges 1 & 2 (UXO #2)  
NSA Mid-South  
Millington, Tennessee





**Legend**

- Proposed Soil Berm Boring  
For PAH and Lead characterization of upper 1/2 of berm. • (1-2', 2-3', 3-4', 4-5', 5-6')
- Proposed Surface/Sub-surface  
Surface (0 - 1') and Subsurface (1-2') sample location for PAH and Lead characterization. Sub-surface "HELD" for analysis pending PAL exceedance in surface soil.
- Proposed Soil Sample  
Surface soil (0-1') sample for PAHs and subsurface soil (1-2') for PAHs and Lead w/ PAH analysis designated for "hold" pending outcome of surface soil.
- Proposed Trenching for PAH and Lead  
Characterization of bottom 1/2 of berm (6-5', 5-4', 4-3', 3-2', 2-1', 1-0', 0-1', -1-2')
- Road
- Composite Sampling Area
- Pistol Range
- Surface Danger Zone
- Target Lines
- Contour (2-ft interval)

**SI Soil Data (TT 2010)**

- FBL Lead Sample Location (No ND)**
  - < 400 mg/kg
  - > 400 mg/kg
- XRF Lead Sample Location (No ND)**
  - < 280 mg/kg
  - > 280 mg/kg



Figure 17-3: Proposed Soil Sampling Locations  
Pistol Range (UXO 1)  
NSA Mid-South  
Millington, Tennessee





### 17.2.1 UXO 2 (Trap/Skeet Ranges #1 and #2)

#### *Soil*

The proposed sample locations shown on Figure 17-1 are intended to close the data gaps and refine the horizontal and vertical extents of PAH and lead contamination identified during the SI (Tt, 2010). Specifically, the following areas will be sampled:

- Areas where elevated PAHs and/or lead were identified in surface soil; however, the vertical extent of PAHs and/or lead needs to be further defined (13 locations)
- Areas in between the east-west SI sampling lines and between lead contaminated and non-contaminated areas (9 locations)
- The three open, grassy areas along the west side of the site (20 locations)
- Behind, or west of, the former shooting stations (11 locations)
- The RV trailer park (2 locations)
- Areas around the Navy Commissary (5 locations)
- The former child development center (4 locations) and adjacent field north of it (2 locations)

Surface soil samples will be collected from 0-1 foot bgs and subsurface soil samples will be collected from 1-2 feet bgs. Most of the subsurface soil samples will be designated for "hold" pending the outcome of the surface soil analyses and whether a PAL exceedance is identified. Most locations are designated for PAH and lead analyses; however, a subset of samples are designated for only one or the other, based on results of the SI and distance from the former firing stations.

Up to 10 additional "discretionary" surface soil samples may be collected based on visual observation of accumulated fragments or lead shot in the areas to be sampled. Additional step-out samples also will be collected beyond the boundaries of the area to be sampled if warranted by lead or PAH concentrations detected in the proposed soil samples.

### ***Sediment***

Eighteen sediment samples are proposed for collection from within the north-south trending drainage ditches and the east-west trending drainage ditch (Figure 17-2) that drain along the east side of both UXO 2 and UXO 1. These ditches receive drainage from the smaller secondary ditches within the shot fall area to the east in the woods. Additionally, PAHs that may be leaching, particularly from the clay pigeons accumulated against the Pistol Range berm, may also accumulate in sediments in the ditches.

Sediment samples will be collected from 0- to 6-inches bgs from 18 sample locations shown on Figure 17-2 and analyzed for lead and PAHs. Samples from the 6 to 12-inch interval also will be collected at the above locations and will be extracted but "held" pending the outcome of the surface sediment interval. Horizontal and vertical step-outs will be collected as warranted, per the criteria discussed in Worksheet 11 and whether exceedances of the PAL (Worksheet #15) exist for the shallow interval.

### **17.2.2 Pistol Range Berm — Test Pits and Soil Borings**

#### ***Soil — Berm Characterization***

Soil below the berm surface (> 1 foot) will be characterized through a combination of hand-auger borings and test pits/excavations. As decided by the BCT and shown on Figure 17-3, the berm will be characterized at eight locations in the front (2), inside (2), and backside (4) of the berm. The upper half of the berm will be characterized with eight soil borings through the top of the berm and sampling five depths in 1-foot intervals, beginning 1 foot below the berm surface (e.g., 1-2 feet, 2-3 feet, 3-4 feet, 4-5 feet, and 5-6 feet). Forty soil samples will be collected with a hand auger from the soil borings for PAH and lead analyses.

The lower half of the berm will be visually inspected and characterized by test pits excavated in the sides of the berm, with sampling resuming at the terminal depth of the soil borings described above. Sampling will proceed from the middle of the berm to its base (i.e., 6-5 feet, 5-4 feet, 4-3 feet, 3-2 feet, 2-1 feet, and 1-0 feet). Since the trap/skeet range operated 7 years before the pistol firing range, the native soil beneath the berm (0-1 feet and 1-2 feet) will also be evaluated for impacts that may have preceded berm construction. Sixty four soil samples will be collected with a backhoe from the eight locations shown on Figure 17-3. Samples will be analyzed for PAHs and lead. Upon completion of the berm's physical assessment and analytical sampling, soil excavated from each test pit will be replaced in the respective pit/trench.



### ***Soil — Firing Stations***

Composite surface and subsurface soil samples (composed of 10 grab samples each) also will be collected from the four areas between the firing line stations shown on Figure 17-3. While the pistol firing line stations were characterized during the SI, the areas between the pistol firing line stations were not. Additionally, the SI soil samples were collected from the 0-1 foot bgs interval only and only analyzed for lead. The additional samples will include subsurface soil samples and PAH analysis in addition to lead since the pistol firing range is within UXO 2.





## SAP WORKSHEET #18: LOCATION-SPECIFIC SAMPLING METHODS/SOP REQUIREMENTS TABLE

(UFP-QAPP Manual Section 3.1.1)

Sample Matrix: Soil (See Figure 17-1 for locations) Sampling Standard Operating Procedure Reference <sup>1</sup> : See Worksheet #21							
Site	Station ID	Sample ID <sup>2</sup>	Depth (feet)	X Coordinate	Y Coordinate	Analytical Group	Number of Samples
UXO 2	1	SRSSB0101	0 — 1	386007.0922	815709.5371	Lead	2
		SRSSB0102	1 — 2			Lead (Hold)	
UXO 2	2	SRSSB0201	0 — 1	385984.4404	815847.8328	Lead	2
		SRSSB0202	1 — 2			Lead (Hold)	
UXO 2	3	SRSSB0301	0 — 1	385859.2589	815831.1418	Lead	2
		SRSSB0302	1 — 2			Lead (Hold)	
UXO 2	4	SRSSB0401	0 — 1	385779.3811	815705.9603	Lead	2
		SRSSB0402	1 — 2			Lead (Hold)	
UXO 2	5	SRSSB0501	0 — 1	385745.9992	815912.2119	Lead	2
		SRSSB0502	1 — 2			Lead (Hold)	
UXO 2	6	SRSSB0601	0 — 1	385656.5837	815797.7604	Lead	2
		SRSSB0602	1 — 2			Lead (Hold)	
UXO 2	7	SRSSB0702	1 — 2	385441.74	815310.8499	Lead, PAHs	1
UXO 2	8	SRSSB0802	1 — 2	385409.1	815713.9502	Lead	1
UXO 2	9	SRSSB0902	1 — 2	385373.79	815896.81	Lead	1
UXO 2	10	SRSSB1001	0 — 1	385284.5458	815178.4798	Lead, PAHs	2
		SRSSB1002	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	11	SRSSB1101	0 — 1	385226.4783	815595.0501	Lead, PAHs	2
		SRSSB1102	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	12	SRSSB1201	0 — 1	385213.855	814953.784	Lead, PAHs	2
		SRSSB1201	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	13	SRSSB1302	1 — 2	385082.4599	815247.3199	Lead, PAHs	1
UXO 2	14	SRSSB1401	0 — 1	385128.9185	815740.9235	Lead	2
		SRSSB1402	1 — 2			Lead	
UXO 2	15	SRSSB1501	0 — 1	385044.7021	814928.5374	Lead, PAHs	2
		SRSSB1502	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	16	SRSSB1601	0 — 1	384997.5096	815113.6994	Lead, PAHs	2
		SRSSB1602	1 — 2			Lead (Hold), PAHs (Hold)	



Sample Matrix: Soil (See Figure 17-1 for locations) Sampling Standard Operating Procedure Reference <sup>1</sup> : See Worksheet #21							
Site	Station ID	Sample ID <sup>2</sup>	Depth (feet)	X Coordinate	Y Coordinate	Analytical Group	Number of Samples
UXO 2	17	SRSSB1701	0 — 1	384971.4112	815185.1264	Lead, PAHs	2
		SRSSB1702	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	18	SRSSB1802	1 — 2	384957.6101	815344.4299	PAHs	1
UXO 2	19	SRSSB1902	1 — 2	384948.78	815662.24	Lead	1
UXO 2	20	SRSSB2002	1 — 2	384895.81	815756.83	Lead	1
UXO 2	21	SRSSB2102	1 — 2	384845.3701	815816.0999	Lead	1
UXO 2	22	SRSSB2201	0 — 1	384797.8919	815640.8082	Lead	2
		SRSSB2202	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	23	SRSSB2301	0 — 1	383995.1701	815112.8401	Lead, PAHs	2
		SRSSB2302	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	24	SRSSB2401	0 — 1	384807.383	815418.3234	Lead, PAHs	2
		SRSSB2402	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	25	SRSSB2501	0 — 1	384901.3579	815135.677	Lead, PAHs	2
		SRSSB2502	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	26	SRSSB2601	0 — 1	384935.6977	815071.1178	Lead, PAHs	2
		SRSSB2602	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	27	SRSSB2701	0 — 1	384873.0246	814910.8648	Lead, PAHs	2
		SRSSB2702	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	28	SRSSB2801	0 — 1	384746.1417	815047.7668	Lead, PAHs	2
		SRSSB2802	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	29	SRSSB2901	0 — 1	384698.8222	814890.6674	Lead, PAHs	2
		SRSSB2902	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	30	SRSSB3002	1 — 2	384653.6701	815118.35	PAHs	1
UXO 2	31	SRSSB3101	0 — 1	384591.4415	815029.6257	Lead, PAHs	2
		SRSSB3102	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	32	SRSSB3201	0 — 1	384575.4084	815140.9132	Lead, PAHs	2
		SRSSB3202	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	33	SRSSB3201	0 — 1	384544.8173	815587.4763	Lead, PAHs	2
		SRSSB3202	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	34	SRSSB3401	0 — 1	384517.046	814867.9453	Lead, PAHs	2
		SRSSB3402	1 — 2			Lead (Hold), PAHs (Hold)	



Sample Matrix: Soil (See Figure 17-1 for locations) Sampling Standard Operating Procedure Reference <sup>1</sup> : See Worksheet #21							
Site	Station ID	Sample ID <sup>2</sup>	Depth (feet)	X Coordinate	Y Coordinate	Analytical Group	Number of Samples
UXO 2	35	SRSSB3501	0 — 1	384496.1868	815034.3413	Lead, PAHs	2
		SRSSB3502	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	36	SRSSB3501	0 — 1	384487.6989	815125.8234	Lead, PAHs	2
		SRSSB3602	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	37	SRSSB3701	0 — 1	384450.9175	815085.2697	Lead, PAHs	2
		SRSSB3702	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	38	SRSSB3801	0 — 1	384417.9084	815024.9102	Lead, PAHs	2
		SRSSB3802	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	39	SRSSB3901	0 — 1	384410.3635	815120.1649	Lead, PAHs	2
		SRSSB3902	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	40	SRSSB4001	0 — 1	384403.436	815395.6014	Lead, PAHs	2
		SRSSB4002	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	41	SRSSB4101	0 — 1	384345.3685	815592.5255	Lead, PAHs	2
		SRSSB4102	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	42	SRSSB4201	0 — 1	384335.7747	814842.6986	Lead, PAHs	2
		SRSSB4202	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	43	SRSSB4301	0 — 1	384344.3455	815019.2514	Lead, PAHs	2
		SRSSB4302	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	44	SRSSB4401	0 — 1	384332.0851	815120.1649	Lead, PAHs	2
		SRSSB4402	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	45	SRSSB4501	0 — 1	384295.3036	815065.4642	Lead, PAHs	2
		SRSSB4502	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	46	SRSSB4601	0 — 1	384260.4084	814999.446	Lead, PAHs	2
		SRSSB4601	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	47	SRSSB4702	1 — 2	384255.3001	815067.42	Lead, PAHs	1
UXO 2	48	SRSSB4801	0 — 1	384241.5459	815095.6436	Lead, PAHs	2
		SRSSB4802	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	49	SRSSB4901	0 — 1	384161.5723	814822.5012	Lead, PAHs	2
		SRSSB4902	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	50	SRSSB5001	0 — 1	384186.8455	815003.2186	Lead, PAHs	2
		SRSSB5002	1 — 2			Lead (Hold), PAHs (Hold)	



Sample Matrix: Soil (See Figure 17-1 for locations) Sampling Standard Operating Procedure Reference <sup>1</sup> : See Worksheet #21							
Site	Station ID	Sample ID <sup>2</sup>	Depth (feet)	X Coordinate	Y Coordinate	Analytical Group	Number of Samples
UXO 2	51	SRSSB5101	0 — 1	384171.1661	815539.5076	Lead, PAHs	2
		SRSSB5102	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	52	SRSSB5201	0 — 1	384004.5378	815554.6555	Lead, PAHs	2
		SRSSB5202	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	53	SRSSB5301	0 — 1	384007.0628	815398.126	Lead, PAHs	2
		SRSSB5302	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	54	SRSSB5401	0 — 1	383968.9861	815062.6349	Lead, PAHs	2
		SRSSB5402	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	55	SRSSB5501	0 — 1	383982.1898	814975.8683	Lead, PAHs	2
		SRSSB5502	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	56	SRSSB5601	0 — 1	383972.2223	814802.304	Lead, PAHs	2
		SRSSB5602	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	57	SRSSB5701	0 — 1	383931.2614	815014.5359	Lead, PAHs	2
		SRSSB5702	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	58	SRSSB5401	0 — 1	383880.3334	814953.2335	Lead, PAHs	2
		SRSSB5402	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	59	SRSSB5401	0 — 1	383871.8452	815043.7724	Lead, PAHs	2
		SRSSB5402	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	60	SRSSB5402	1 — 2	383841.1881	815083.2309	Lead, PAHs	1
UXO 2	61	SRSSB5401	0 — 1	383848.0083	815645.5438	Lead, PAHs	2
		SRSSB6101	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	62	SRSSB6201	0 — 1	383798.0199	814779.5819	Lead, PAHs	2
		SRSSB6202	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	63	SRSSB6301	0 — 1	383716.6299	815037.9699	Lead, PAHs	2
		SRSSB6302	1 — 2			Lead, PAHs	
UXO 2	64	SRSSB6402	1 — 2	383673.468	814948.89	PAHs	1
UXO 2	65	SRSSB6501	0 — 1	383621.2932	814761.9091	Lead, PAHs	2
		SRSSB6502	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	66	SRSSB6601	0 — 1	383459.2092	814734.1378	Lead, PAHs	2
		SRSSB6602	1 — 2			Lead (Hold), PAHs (Hold)	
UXO 2	Field Duplicate	TBD <sup>3</sup>	TBD <sup>3</sup>	TBD <sup>3</sup>	TBD <sup>3</sup>	Lead	4



Sample Matrix: Soil (See Figure 17-1 for locations) Sampling Standard Operating Procedure Reference <sup>1</sup> : See Worksheet #21							
Site	Station ID	Sample ID <sup>2</sup>	Depth (feet)	X Coordinate	Y Coordinate	Analytical Group	Number of Samples
UXO 2	Field Duplicate	TBD <sup>3</sup>	TBD <sup>3</sup>	TBD <sup>3</sup>	TBD <sup>3</sup>	PAHs	3
UXO 2	Field Duplicate	TBD <sup>3</sup>	TBD <sup>3</sup>	TBD <sup>3</sup>	TBD <sup>3</sup>	Lead (Hold), PAHs (Hold)	3
UXO 2	MS/MSD <sup>4</sup>	TBD <sup>3</sup>	TBD <sup>3</sup>	TBD <sup>3</sup>	TBD <sup>3</sup>	Lead	4
UXO 2	MS/MSD <sup>4</sup>	TBD <sup>3</sup>	TBD <sup>3</sup>	TBD <sup>3</sup>	TBD <sup>3</sup>	PAHs	3
UXO 2	MS/MSD <sup>4</sup>	TBD <sup>3</sup>	TBD <sup>3</sup>	TBD <sup>3</sup>	TBD <sup>3</sup>	Lead (Hold), PAHs (Hold)	3
UXO 2	Equipment Blank	EBMMDDYY	not applicable	not applicable	not applicable	Lead, PAHs	2

**Notes:**

<sup>1</sup> Standard operating procedure or worksheet that describes the sample collection procedures

<sup>2</sup> Sample identification (ID) will be the station ID and the bottom depth, to be determined in the field.

<sup>3</sup> To be determined. Field duplicates and sample depths will be determined in the field.

<sup>4</sup> Samples used for matrix spike/matrix spike duplicates will be determined in the field and will use the same sample ID appended with an MS or MSD

Coordinates are approximate and final coordinates will be determined in the field.

PAHs = Polynuclear aromatic hydrocarbons

MS/MSD = Matrix spike/matrix spike duplicate

Frequency of QA/QC sample collection:

Duplicate — One per 20 field samples

Matrix spike/matrix spike duplicate — One pair per 20 field samples (including field QC samples)

Equipment Blank — One per week of sampling



Sample Matrix: Sediment (See Figure 17-2 for locations) Sampling Standard Operating Procedure Reference <sup>1</sup> : See Worksheet #21							
Site	Station ID	Sample ID	Depth (inches)	X Coordinates	Y Coordinates	Analytical Group	Number of Samples
UXO 2	1	SRM00100	0 — 6	384994.8449	815377.6769	Lead, PAHs	2
		SRM00101	6 — 12			Lead (Hold), PAHs (Hold)	
UXO 2	2	SRM00200	0 — 6	384964.1314	815120.1554	Lead, PAHs	2
		SRM00201	6 — 12			Lead (Hold), PAHs (Hold)	
UXO 2	3	SRM00300	0 — 6	384836.5516	815110.705	Lead, PAHs	2
		SRM00301	6 — 12			Lead (Hold), PAHs (Hold)	
UXO 2	4	SRM00400	0 — 6	384619.1941	815051.6405	Lead, PAHs	2
		SRM00401	6 — 12			Lead (Hold), PAHs (Hold)	
UXO 2	5	SRM00500	0 — 6	384569.5797	815306.7994	Lead, PAHs	2
		SRM00501	6 — 12			Lead (Hold), PAHs (Hold)	
UXO 2	6	SRM00600	0 — 6	384524.6907	815557.2333	Lead, PAHs	2
		SRM00601	6 — 12			Lead (Hold), PAHs (Hold)	
UXO 2	7	SRM00700	0 — 6	384493.9771	815819.4798	Lead, PAHs	2
		SRM00701	6 — 12			Lead (Hold), PAHs (Hold)	
UXO 2	8	SRM00800	0 — 6	384460.9011	815294.9864	Lead, PAHs	2
		SRM00801	6 — 12			Lead (Hold), PAHs (Hold)	
UXO 2	9	SRM00900	0 — 6	384368.7602	815538.3324	Lead, PAHs	2
		SRM00900	6 — 12			Lead (Hold), PAHs (Hold)	
UXO 2	10	SRM01000	0 — 6	384278.9822	815784.0412	Lead, PAHs	2
		SRM01001	6 — 12			Lead (Hold), PAHs (Hold)	
UXO 2	11	SRM01100	0 — 6	384430.1872	815028.0145	Lead, PAHs	2
		SRM01101	6 — 12			Lead (Hold), PAHs (Hold)	
UXO 2	12	SRM01200	0 — 6	384392.3861	815179.2199	Lead, PAHs	2
		SRM01201	6 — 12			Lead (Hold), PAHs (Hold)	
UXO 2	13	SRM01300	0 — 6	384271.8942	815006.7511	Lead, PAHs	2
		SRM01301	6 — 12			Lead (Hold), PAHs (Hold)	
UXO 2	14	SRM01400	0 — 6	384248.2686	815127.243	Lead, PAHs	2
		SRM01401	6 — 12			Lead (Hold), PAHs (Hold)	
UXO 2	15	SRM01500	0 — 6	383983.6593	814976.0376	Lead, PAHs	2
		SRM01501	6 — 12			Lead (Hold), PAHs (Hold)	
UXO 2	16	SRM01600	0 — 6	383733.2254	814950.0491	Lead, PAHs	2





Sample Matrix: Sediment (See Figure 17-2 for locations) Sampling Standard Operating Procedure Reference <sup>1</sup> : See Worksheet #21							
Site	Station ID	Sample ID	Depth (inches)	X Coordinates	Y Coordinates	Analytical Group	Number of Samples
UXO 2	17	SRM01601	6 — 12	383714.3248	815117.7926	Lead (Hold), PAHs (Hold)	2
		SRM01700	0 — 6			Lead, PAHs	
		SRM01701	6 — 12			Lead (Hold), PAHs (Hold)	
UXO 2	18	SRM01800	0 — 6	383572.5695	814919.3356	Lead, PAHs	2
		SRM01801	6 — 12			Lead (Hold), PAHs (Hold)	
UXO 2	Field Duplicate	TBD <sup>2</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	Lead, PAHs	1
UXO 2	Field Duplicate	TBD <sup>2</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	Lead (Hold), PAHs (Hold)	1
UXO 2	MS/MSD <sup>3</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	Lead, PAHs	1
UXO 2	MS/MSD <sup>3</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	Lead (Hold), PAHs (Hold)	1
UXO 2	Equipment Blank	EBMMDDYY	not applicable	not applicable	not applicable	Lead, PAHs	1

**Notes:**

<sup>1</sup> Standard operating procedure or worksheet that describes the sample collection procedures

<sup>2</sup> To be determined. Field duplicates and sample depths will be determined in the field.

<sup>3</sup> Samples used for matrix spike/matrix spike duplicates will be determined in the field and will use the same sample ID appended with an MS or MSD

Coordinates are approximate and final coordinates will be determined in the field.

PAHs = Polynuclear aromatic hydrocarbons

MS/MSD = Matrix spike/matrix spike duplicate

Frequency of QA/QC sample collection:

Field Duplicate — One per 20 field samples

Matrix spike/matrix spike duplicate — One pair per 20 field samples (including field QC samples)

Equipment Blank — One per week of sampling



<b>Sample Matrix: Soil (See Figure 17-3 for locations)</b> <b>Sampling Standard Operating Procedure Reference<sup>1</sup>: See Worksheet #21</b>							
Site	Station ID	Sample ID	Depth (feet)	X Coordinates	Y Coordinates	Analytical Group	Number of Samples
UXO 1	1	PRSSB01-depth	1-2 (Hand Auger)	384192.4656	815004.2882	Lead, PAHs	13
			2-3 (Hand Auger)				
			3-4 (Hand Auger)				
			4-5 (Hand Auger)				
			5-6 (Hand Auger)				
			6-7 (Trenching)				
			7-8 (Trenching)				
			8-9 (Trenching)				
			9-10 (Trenching)				
			10-11 (Trenching)				
			11-12 (Trenching)				
			12-13 (Trenching; 0-1 below grade)				
			13-14 (Trenching; 1-2 below grade)				
UXO 1	2	PRSSB02-depth	1-2 (Hand Auger)	384174.6809	815062.228	Lead, PAHs	13
			2-3 (Hand Auger)				
			3-4 (Hand Auger)				
			4-5 (Hand Auger)				
			5-6 (Hand Auger)				
			6-7 (Trenching)				
			7-8 (Trenching)				
			8-9 (Trenching)				
			9-10 (Trenching)				
			10-11 (Trenching)				
			11-12 (Trenching)				
			12-13 (Trenching; 0-1 below grade)				
			13-14 (Trenching; 1-2 below grade)				
UXO 1	3	PRSSB03-depth	1-2 (Hand Auger)	384139.6691	815106.5521	Lead, PAHs	13
			2-3 (Hand Auger)				
			3-4 (Hand Auger)				
			4-5 (Hand Auger)				
			5-6 (Hand Auger)				
			6-7 (Trenching)				



Sample Matrix: Soil (See Figure 17-3 for locations) Sampling Standard Operating Procedure Reference <sup>1</sup> : See Worksheet #21							
Site	Station ID	Sample ID	Depth (feet)	X Coordinates	Y Coordinates	Analytical Group	Number of Samples
UXO 1	3	PRSSB03-depth	7-8 (Trenching)	384139.6691	815106.5521	Lead, PAHs	13
			8-9 (Trenching)				
			9-10 (Trenching)				
			10-11 (Trenching)				
			11-12 (Trenching)				
			12-13 (Trenching; 0-1 below grade)				
			13-14 (Trenching; 1-2 below grade)				
UXO 1	4	PRSSB04-depth	1-2 (Hand Auger)	384111.1862	815095.3805	Lead, PAHs	13
			2-3 (Hand Auger)				
			3-4 (Hand Auger)				
			4-5 (Hand Auger)				
			5-6 (Hand Auger)				
			6-7 (Trenching)				
			7-8 (Trenching)				
			8-9 (Trenching)				
			9-10 (Trenching)				
			10-11 (Trenching)				
			11-12 (Trenching)				
			12-13 (Trenching; 0-1 below grade)				
			13-14 (Trenching; 1-2 below grade)				
UXO 1	5	PRSSB05-depth	1-2 (Hand Auger)	384068.1319	815087.1164	Lead, PAHs	13
			2-3 (Hand Auger)				
			3-4 (Hand Auger)				
			4-5 (Hand Auger)				
			5-6 (Hand Auger)				
			6-7 (Trenching)				
			7-8 (Trenching)				
			8-9 (Trenching)				
			9-10 (Trenching)				
			10-11 (Trenching)				
			11-12 (Trenching)				
			12-13 (Trenching; 0-1 below grade)				
			13-14 (Trenching; 1-2 below grade)				



<b>Sample Matrix: Soil (See Figure 17-3 for locations)</b> <b>Sampling Standard Operating Procedure Reference<sup>1</sup>: See Worksheet #21</b>							
Site	Station ID	Sample ID	Depth (feet)	X Coordinates	Y Coordinates	Analytical Group	Number of Samples
UXO 1	6	PRSSB06-depth	1-2 (Hand Auger)	384032.5824	815083.9678	Lead, PAHs	13
			2-3 (Hand Auger)				
			3-4 (Hand Auger)				
			4-5 (Hand Auger)				
			5-6 (Hand Auger)				
			6-7 (Trenching)				
			7-8 (Trenching)				
			8-9 (Trenching)				
			9-10 (Trenching)				
			10-11 (Trenching)				
			11-12 (Trenching)				
			12-13 (Trenching; 0-1 below grade)				
			13-14 (Trenching; 1-2 below grade)				
UXO 1	7	PRSSB07-depth	1-2 (Hand Auger)	384041.1024	815013.4378	Lead, PAHs	13
			2-3 (Hand Auger)				
			3-4 (Hand Auger)				
			4-5 (Hand Auger)				
			5-6 (Hand Auger)				
			6-7 (Trenching)				
			7-8 (Trenching)				
			8-9 (Trenching)				
			9-10 (Trenching)				
			10-11 (Trenching)				
			11-12 (Trenching)				
			12-13 (Trenching; 0-1 below grade)				
			13-14 (Trenching; 1-2 below grade)				
UXO 1	8	PRSSB008-depth	1-2 (Hand Auger)	384037.3786	814956.0941	Lead, PAHs	13
			2-3 (Hand Auger)				
			3-4 (Hand Auger)				
			4-5 (Hand Auger)				
			5-6 (Hand Auger)				
			6-7 (Trenching)				



Sample Matrix: Soil (See Figure 17-3 for locations) Sampling Standard Operating Procedure Reference <sup>1</sup> : See Worksheet #21							
Site	Station ID	Sample ID	Depth (feet)	X Coordinates	Y Coordinates	Analytical Group	Number of Samples
UXO 1	8	PRSSB008-depth	7-8 (Trenching)	384037.3786	814956.0941	Lead, PAHs	13
			8-9 (Trenching)				
			9-10 (Trenching)				
			10-11 (Trenching)				
			11-12 (Trenching)				
			12-13 (Trenching; 0-1 below grade)				
			13-14 (Trenching; 1-2 below grade)				
UXO 1	9	PRSSB00901	0-1	384149.0366	814958.933	Lead, PAHs	2
		PRSSB00902	1-2			Lead (Hold), PAHs (Hold)	
UXO 1	10	PRSSB01001	0-1	384144.3053	814986.3742	Lead, PAHs	2
		PRSSB01002	1-2			Lead (Hold), PAHs (Hold)	
UXO 1	11	PRSSB01101	0-1	384100.7775	814978.8043	Lead, PAHs	2
		PRSSB01102	1-2			Lead (Hold), PAHs (Hold)	
UXO 1	12	PRSSB01201	0-1	384105.5088	814953.2555	Lead, PAHs	2
		PRSSB01202	1-2			Lead (Hold), PAHs (Hold)	
UXO 1	13	PRSSB01301	0-1	384204.6299	815099.7099	PAHs	2
		PRSSB01302	1-2			Lead, PAHs (Hold)	
UXO 1	14	PRSSB01401	0-1	384186.69	815115.39	PAHs	2
		PRSSB01401	1-2			Lead, PAHs (Hold)	
UXO 1	15	PRSSB01502	0-1	384126.4798	815136.0599	PAHs	2
		PRSSB01501	1-2			Lead, PAHs (Hold)	
UXO 1	16	PRSSB01602	0-1	384113.7899	815148.4998	PAHs	2
		PRSSB01601	1-2			Lead, PAHs (Hold)	
UXO 1	17	PRSSB01702	0-1	384072.52	815126.6898	PAHs	2
		PRSSB01701	1-2			Lead, PAHs (Hold)	
UXO 1	18	PRSSB01802	0-1	384068.5	815145.78	PAHs	2
		PRSSB01801	1-2			Lead, PAHs (Hold)	
UXO 1	19	PRSSB01902	0-1	383995.1701	815112.8401	PAHs	2
		PRSSB01901	1-2			Lead, PAHs (Hold)	
UXO 1	Field Duplicate	TBD <sup>2</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	Lead, PAHs	6





Sample Matrix: Soil (See Figure 17-3 for locations) Sampling Standard Operating Procedure Reference <sup>1</sup> : See Worksheet #21							
Site	Station ID	Sample ID	Depth (feet)	X Coordinates	Y Coordinates	Analytical Group	Number of Samples
UXO 1	Field Duplicate	TBD <sup>2</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	Lead (Hold), PAHs (Hold)	2
UXO 1	MS/MSD <sup>3</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	Lead, PAHs	6
UXO 1	MS/MSD <sup>3</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	TBD <sup>2</sup>	Lead (Hold), PAHs (Hold)	2
UXO 1	Equipment Blank	EBMMDDYY	not applicable	not applicable	not applicable	Lead, PAHs	2

**Notes:**

<sup>1</sup> Standard operating procedure or worksheet that describes the sample collection procedures

<sup>2</sup> To be determined. Field duplicates and sample depths will be determined in the field.

<sup>3</sup> Samples used for matrix spike/matrix spike duplicates will be determined in the field and will use the same sample ID appended with an MS or MSD

Coordinates are approximate and final coordinates will be determined in the field.

PAHs = Polynuclear aromatic hydrocarbons

MS/MSD = Matrix spike/matrix spike duplicate

Frequency of QA/QC sample collection:

Field Duplicate — One per 20 field samples

Matrix spike/matrix spike duplicate — One pair per 20 field samples (including field QC samples)

Equipment Blank — One per week of sampling



## SAP WORKSHEET #19: ANALYTICAL METHODS/SOP REQUIREMENTS TABLE

(UFP-QAPP Manual Section 3.1.1)

Laboratory Name and Address: GCAL, 7979 GSRI Road, Baton Rouge, LA 70820  
Laboratory Point of Contact/Project Manager: Shelley Bourgeois, [shelley.bourgeois@gcal.com](mailto:shelley.bourgeois@gcal.com), (225) 214-7077

Matrix	Analytical Group	Analytical and Preparation Method/ GCAL SOP Reference	Containers (number, size, and type)	Sample Volume	Preservation Requirements <sup>1</sup>	Maximum Holding Time <sup>2</sup> (preparation/analysis)
Soil and Sediment	PAHs via SIM	SW-846 3550C/8270D EXT-001/GCMSSV-004	(1) 4 ounce glass jar	30 grams	Cool to 0-6°C	14 days to preparation 40 days from preparation to analysis
Soil and Sediment	Lead	SW-846 3050B/6020A MET-004/MET-021	(1) 4 ounce glass jar	1.25 grams	None	180 days

### Notes:

<sup>1</sup> Chemical, temperature, and light protected

<sup>2</sup> Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted

GCAL = Gulf Coast Analytical Laboratory  
SOP = Standard operating procedure  
PAHs = Polynuclear aromatic hydrocarbons  
SIM = Selective ion monitoring  
°C = Degrees Celsius



## SAP WORKSHEET #20: FIELD QUALITY CONTROL SAMPLE SUMMARY TABLE

(UFP-QAPP Manual Section 3.1.1)

Site	Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates	No. of MS/MSDs <sup>1</sup>	No. of Equipment Blanks	Total No. of Samples to Lab
UXO 2	Soil	PAHs	53	3	3/3	1	57
UXO 2	Soil	Lead	65	4	4/4	1	70
UXO 2	Soil	PAHs (Hold)	47	3	3/3	1	51
UXO 2	Soil	Lead (Hold)	53	3	3/3	1	57
UXO 2	Sediment	PAHs	18	1	1/1	1	20
UXO 2	Sediment	PAHs (Hold)	18	1	1/1	1	20
UXO 2	Sediment	Lead (Hold)	18	1	1/1	1	20
UXO 2	Sediment	Lead	18	1	1/1	1	20
UXO 1	Soil	PAHs	126	6	6/6	1	133
UXO 1	Soil	Lead	126	6	6/6	1	133
UXO 1	Soil	PAHs (Hold)	22	2	2/2	1	25
UXO 1	Soil	Lead (Hold)	8	1	1/1	1	10

### Notes:

<sup>1</sup> Although MS/MSD samples are not typically considered field QC samples, they are included here because location determination is often established in the field. MS/MSD samples are not included in the total number of samples sent to the laboratory. For total metals, an MD will be collected in place of an MSD.  
MS/MSD = Matrix spike/matrix spike duplicate



## SAP WORKSHEET #21: PROJECT SAMPLING SOP REFERENCES TABLE

*(UFP-QAPP Manual Section 3.1.2)*

Field Standard Operating Procedure Reference Table					
SOP Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Yes/No)	Comments
SOP-3-01	Utility Clearance, Revision 0; June 2012	Resolution Consultants	None	No	See Appendix A
SOP-3-02	Field Log Books, Revision 0; May 2012	Resolution Consultants	None	No	See Appendix A
SOP-3-03A	Sample Labeling and Chain of Custody, Revision 0; August 2012	Resolution Consultants	None	No	See Appendix A
SOP-3-04A	Packaging and Shipping Procedures for Low Concentration Samples, Revision 0; May 2012	Resolution Consultants	None	No	See Appendix A
SOP-3-05 (MS)	Investigative Derived Waste Management, Revision 0; August 2012	Resolution Consultants	None	No	See Appendix A
SOP-3-6	Equipment Decontamination, Revision 0; May 2012	Resolution Consultants	Buckets, brushes	No	See Appendix A
SOP-3-08 (MS)	Soil Classification, Revision 0; May 2012	Resolution Consultants	None	No	See Appendix A
SOP-3-21	Surface and Subsurface Soil Sampling, Revision 0; May 2012	Resolution Consultants	Hand auger	No	See Appendix A
SOP-3-22	Sediment Sampling, Revision 0; May 2012	Resolution Consultants	Hand auger	No	See Appendix A

**Note:**  
SOP = Standard operating procedure



## SAP WORKSHEET #22: FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION TABLE

(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment Calibration, Maintenance, Testing, and Inspection Table							
Field Equipment	Activity <sup>1</sup>	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference	Comments
Schonstedt, Whites, or equivalent detector(s)	Visual Inspection Testing and Maintenance	Twice daily, at the beginning and end of each working day	Test instrument detection abilities with metallic objects; only use instrument if metallic objects are detected	Operator correction or replacement	Resolution Consultants UXO Technician	NA	None

**Notes:**

<sup>1</sup> Activities may include calibration, verification, testing, maintenance, and/or inspection.

SOP = Standard operating procedure

NA = not applicable





## SAP WORKSHEET #23: ANALYTICAL SOP REFERENCES TABLE

(UFP-QAPP Manual Section 3.2.1)

<b>Laboratory Name and Address:</b> GCAL, 7979 GSRI Road, Baton Rouge, LA 70820 <b>Laboratory Point of Contact/Project Manager:</b> Shelley Bourgeois, <a href="mailto:shelley.bourgeois@gcal.com">shelley.bourgeois@gcal.com</a> , (225) 214-7077						
GCAL SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Variance to QSM	Modified for Project Work? (Yes/No)
GCMSSV-004	<i>SOP for the Analysis of Method 8270D</i> , Revision 7, 2/13/12	Definitive	Aqueous and Soil Analysis PAHs via SIM	GC/MS Agilent 6890/5973 and Agilent 7890/5975	No	No
EXT-003	<i>SOP for BNA by Separatory Funnel</i> , Revision 21, 11/18/11	Definitive	Aqueous Extraction PAHs via SIM	Not applicable	No	No
EXT-001	<i>SOP for BNA Solids</i> , Revision 18, 11/18/11	Definitive	Soil Extraction PAHs via SIM	Not applicable	No	No
MET-021	<i>SOP for the Analysis of Samples by ICP/MS</i> , 7/15/11, Revision 2	Definitive	Aqueous and Soil Analysis Lead	Agilent 7700	No	No
MET-005	<i>SOP for Metals Aqueous Preparation</i> , Revision 14, 3/28/11	Definitive	Aqueous Digestion Lead	Not applicable	No	No
MET-004	<i>SOP for Metals Soil Digestion</i> , Revision 14, 3/28/11	Definitive	Soil Digestion Lead	Not applicable	No	No

### Notes:

GCAL = Gulf Coast Analytical Laboratory  
 SOP = Standard operating procedure  
 QSM = Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, October 2010 (and subsequent revisions)  
 PAH = Polynuclear aromatic hydrocarbons  
 SIM = Selective ion monitoring  
 GC/MS = Gas chromatograph/mass spectrometer  
 GC/ECD = Gas chromatography/electron capture detector  
 ICP/MS = Inductively coupled plasma spectroscopy/mass spectrometer



## SAP WORKSHEET #24: ANALYTICAL INSTRUMENT CALIBRATION TABLE

(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	GCAL SOP Reference
GC/MS PAHs via SIM	Breakdown check (DDT				Analyst, Supervisor	GCAL SOP GCMSSV-004
GC/MS PAHs via SIM	CCV	CV daily, before sample analysis, and every 12 hours of analysis time	All targets $\leq 20$ %D	Repeat initial calibration and reanalyze all samples analyzed since the last successful CV.	Analyst, Supervisor	GCAL SOP GCMSSV-004
GC/MS PAHs via SIM	ICAL Minimum five-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD $\leq 30$ for RFs of the CCCs; Average %RSD $\leq 15\%$ for all compounds, linear or quadratic curve fit with COD $\geq 0.99$	Repeat calibration if criterion is not met	Analyst, Supervisor	GCAL SOP GCMSSV-004
GC/MS PAHs via SIM	Second source CV	Once after each ICAL	All analytes within $\pm 20\%$ of expected value	Remake standard, recalibrate if necessary	Analyst, Supervisor	GCAL SOP GCMSSV-004
GC/MS PAHs via SIM	RRT Evaluation	Prior to sample analysis	Set at mid-point of ICAL; $\pm 30$ seconds each CCV	CCV fails, perform column maintenance, inspect pumps, and leak checks	Analyst, Supervisor	GCAL SOP GCMSSV-004
ICP/MS Lead	ICAL for all analytes: minimum one high standard and a calibration blank	Daily initial calibration prior to sample analysis	If more than one calibration standard is used, $r^2 \geq 0.995$	Recalibrate and/or perform necessary equipment maintenance.	Analyst, Supervisor	GCAL SOP MET-021
ICP/MS Lead	Second source ICV	Once after each initial calibration	All analytes within $\pm 10\%$ of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification.	Analyst, Supervisor	GCAL SOP MET-021
ICP/MS Lead	CCV	CCV after every 10 samples and at the end of the analytical sequence	All analytes within $\pm 10\%$ of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification.	Analyst, Supervisor	GCAL SOP MET-021
ICP/MS Lead	Low-level calibration check standard	Daily following calibration	80-120% recovery	Recalibrate and/or perform necessary equipment maintenance.	Analyst, Supervisor	GCAL SOP MET-021



## SAP WORKSHEET #24: ANALYTICAL INSTRUMENT CALIBRATION TABLE (Continued)

(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	GCAL SOP Reference
ICP/MS Lead	Calibration blank	Once with each ICAL, after every 10 samples and at the end of an analytical sequence.	Determined concentration $\leq$ LOD	Determine source of possible contamination, perform maintenance and recalibrate.	Analyst, Supervisor	GCAL SOP MET-021
ICP/MS Lead	Linear Dynamic range/ High level check	Every 6 months and with major maintenance	90-110% recovery	Perform maintenance and/or reanalyze at lower concentration.	Analyst, Supervisor	GCAL SOP MET-021

### Notes:

GCAL	=	Gulf Coast Analytical Laboratory
SOP	=	Standard operating procedure
GC/MS	=	Gas chromatograph/mass spectrometer
PAH	=	Polynuclear aromatic hydrocarbons
SIM	=	Selective ion monitoring
DDT	=	4,4'-dichlorodiphenyltrichloroethane
CCV	=	Continuing calibration verification
CV	=	Calibration verification
%D	=	Percent difference
ICAL	=	Initial calibration
%RSD	=	Relative standard deviation
RFs	=	Response factors
CCCs	=	Calibration check compounds
COD	=	Coefficient of the determination
RRT	=	Relative retention times
r <sup>2</sup>	=	Correlation coefficient
ICV	=	Initial calibration verification
ICP/MS	=	Inductively coupled plasma spectroscopy/mass spectrometer
LOD	=	Limit of detection



## SAP WORKSHEET #25: ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION TABLE

(UFP-QAPP Manual Section 3.2.3)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	GCAL SOP Reference
GC/MS	Check for leaks, replace gas line filters, replace column, clean injection port/liner	PAHs via SIM	Monitor instrument performance via Continuing Calibration Verification	As needed	No maintenance is required as long as instrument QC meets DoD QSM criteria	Replace connections, clean source, replace gas line filters, replace GC column, clip column, replace injection port liner, clean injection port, replace Electron Multiplier	Analyst, Supervisor	GCAL SOP GCMSSV-004
ICP/MS	Change pump tubing, clean nebulizer, change torch, clean sample cone/skimmer cone	Lead	Monitor instrument performance via Continuing Calibration Verification and Continuing Calibration Blank	As needed	No maintenance is required as long as instrument QC meets DoD QSM criteria	Change pump tubing, change torch and nebulizer, clean cone; recalibrate and reanalyze affected data	Analyst, Supervisor	GCAL SOP MET-021

### Notes:

GCAL	=	Gulf Coast Analytical Laboratory
SOP	=	Standard operating procedure
GC/MS	=	Gas chromatograph/mass spectrometer
PAHs	=	Polynuclear aromatic hydrocarbons
SIM	=	Selective ion monitoring
QC	=	Quality control
DoD	=	Department of Defense
QSM	=	Quality Systems Manual
GC	=	Gas chromatography
ICP/MS	=	Inductively coupled plasma spectroscopy/mass spectrometer



## SAP WORKSHEET #26: SAMPLE HANDLING SYSTEM

(UFP-QAPP Manual Appendix A)

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT	
Sample Collection (Personnel/Organization):	FTL/Resolution Consultants
Sample Packaging (Personnel/Organization):	FTL/Resolution Consultants
Coordination of Shipment (Personnel/Organization):	FTL/Resolution Consultants
Type of Shipment/Carrier:	Overnight via FedEx
SAMPLE RECEIPT AND ANALYSIS	
Sample Receipt (Personnel/Organization):	Sample Receiving Supervisor, Charlotte Saucier/GCAL
Sample Custody and Storage (Personnel/Organization):	Sample Receiving Supervisor, Charlotte Saucier/GCAL
Sample Preparation (Personnel/Organization):	Extractions, Prep Supervisor, Rob Martin/GCAL
Sample Determinative Analysis (Personnel/Organization):	Laboratory Manager, Scott Bailey/GCAL
SAMPLE ARCHIVING	
Field Sample Storage (No. of days from sample collection):	180 Days from Receipt of Samples
Sample Extract/Digestate Storage (No. of days from extraction/digestion):	180 Days from Receipt of Samples
SAMPLE DISPOSAL	
Personnel/Organization:	Waste Compliance Manager John Bailey/GCAL
Number of Days from Analysis:	180 Days from Receipt of Samples

### Notes:

FTL = Field Team Leader  
GCAL = Gulf Coast Analytical Laboratories



## **SAP WORKSHEET #27: SAMPLE CUSTODY REQUIREMENTS**

*(UFP-QAPP Manual Section 3.3.3)*

### **27.1 Sample Nomenclature, Sample Collection Documentation, Handling, and Tracking Procedures**

The following sections outline the procedures that will be used to document project activities and sample collection, handling, tracking, and custody procedures during the investigation. All forms must be filled in as completely as possible.

#### **27.1.1 Sample Nomenclature**

Sample labeling will be conducted in general accordance with the procedures outlined in Worksheet #18. Nomenclature for solid (soil) samples includes the site being investigated, soil boring identification number, sample media, sample depth, and sample type code (i.e., normal, field duplicate, equipment blank, etc.). Nomenclature for aqueous samples includes the site being investigated, monitoring well identification number, sample media, sample date, and sample type code (i.e., normal, field duplicate, equipment blank, etc.). Sample type codes planned for this event will include N for normal samples, FD for field duplicates, and EB for equipment blanks. Equipment blanks will be labeled sequentially followed by the date (i.e., EB20131213,). Samples to be used for MS and MSD will be labeled MS/MSD on the container label and noted on the chain-of-custody; however, "MS/MSD" will not be part of the unique sample identifier in order to maintain consistency with the project database. Worksheet #18 provides anticipated sample identifiers for this scope of work.

#### **27.1.2 Sample Collection Documentation**

Documentation of field observations will be recorded in a field logbook(s) and/or field log sheets including sample collection logs, boring logs, and monitoring well construction logs. The field logbook utilized on this project will consist of a bound, water-resistant logbook. All pages of the logbook will be numbered sequentially and observations will be recorded with indelible ink.

Field sample log sheets will be used to document sample collection details and other observations and activities will be recorded in the field logbook. Instrument calibration logs will be used to record the daily instrument calibration.

For sampling and field activities, the following types of information will be recorded in the field logbook as appropriate:

- Site name and location
- Date and time of logbook entries
- Personnel and their affiliations
- Weather conditions
- Activities involved with the sampling
- Subcontractor activity summary
- Site observations including site entry and exit times
- Site sketches made on site
- Visitor names, affiliations, arrival and departure times
- Health and safety issues, including personal protective equipment

### **27.1.3 Sample Handling and Tracking System**

Following collection, all samples will be immediately placed on ice in a cooler. The glass sample containers will be enclosed in bubble-wrap in order to protect the bottle ware during shipment. The cooler will be secured using strapping tape along with a signed custody seal. Sample coolers will be delivered to a local courier location for priority overnight delivery to the selected laboratory for analysis. Samples will be preserved as appropriate based on the analytical method. The laboratories will provide pre-preserved sample containers for sample collection. Samples will be maintained at 0 to 6 degrees Celsius (°C) until delivery to the laboratory. Proper custody procedures will be followed throughout all phases of sample collection and handling.

After collection, each sample will be maintained in the sampler's custody until formally transferred to another party (e.g., FedEx). For all samples collected, chain-of-custody forms will document the date and time of sample collection, the sampler's name, and the names of all others who subsequently held custody of the sample. Specifications for chemical analyses will also be documented on the chain-of-custody form. Further details on chain-of-custody procedures are provided in SOP-3-03A.

The following subsections outline the procedures that will be used by field and laboratory personnel to document project activities and sample-collection procedures. All forms must be filled in as completely as possible.

Resolution Consultants personnel will collect the samples. The samplers will take care not to contaminate samples through improper handling. Samples will be sealed in appropriate containers, packaged by Resolution Consultants' personnel and placed into sealed coolers under chain-of-

custody in accordance with SOP-3-03A. All coolers will contain a temperature blank. Samples will be transferred under chain-of-custody to a courier as described below. Once received by the laboratory, receipt will be documented on the chain-of-custody form and the samples will be checked in. The samples will remain under chain-of-custody throughout the analysis period to ensure their integrity is preserved. Details are provided below.

Samples to be delivered to the laboratory(s) will be made by a public courier (i.e., FedEx). After samples have been collected, they will be sent to the laboratory(s) within 24 hours. Under no circumstances will sample holding times be exceeded.

## **27.2 Field Sample Custody Procedures**

Chain-of-custody protocols will be used throughout sample handling to establish the evidentiary integrity of sample containers. These protocols will be used to demonstrate that the samples were handled and transferred in a manner that would eliminate possible tampering. Samples for the laboratory will be packaged and shipped in accordance with SOP-3-04A.

A sample is under custody if:

- The sample is in the physical possession of an authorized person
- The sample is in view of an authorized person after being in his/her possession
- The sample is placed in a secure area by an authorized person after being in his/her possession
- The sample is in a secure area, restricted to authorized personnel only

Custody documentation is designed to provide documentation of preparation, handling, storage, and shipping of all samples collected. A multi-part form is used with each page of the form signed and dated by the recipient of a sample or portion of sample. The person releasing the sample and the person receiving the sample each will retain a copy of the form each time a sample transfer occurs.



Integrity of the samples collected will be the responsibility of identified persons from the time the samples are collected until the samples, or their derived data, are incorporated into the final report.

The Resolution Consultants FTL is responsible for the care and custody of the samples collected until they are delivered to the laboratory or are entrusted to a carrier. When transferring samples, the individuals relinquishing and receiving them will sign, date, and note the time on the chain-of-custody form. This record documents the sample custody transfer from the sampler to the laboratory, often through another person or agency (common carrier). Upon arrival at the laboratory, internal sample custody procedures will be followed as defined in the Laboratory SOPs (Appendix B).

### **27.3 Laboratory Chain-of-Custody**

Laboratory sample custody procedures (receipt of samples, archiving, and disposal) will be used per SOPs in Appendix A. Coolers are received and checked for proper temperature. A sample cooler receipt form will be filled out to note conditions and any discrepancies. The chain-of-custody form will be checked against the sample containers for accuracy. Samples will be logged into the Laboratory Information Management System and given a unique log number which can be tracked through processing. The Laboratory PM will notify the Resolution Consultants FTL verbally or via e-mail of any problems on the same day that an issue is identified.



## SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE

(UFP-QAPP Manual Section 3.4)

Matrix		Sediment and Soil				
Analytical Group		PAHs via SIM				
Analytical Method		SW-846 8270D				
SOP Reference		GCAL SOP GCMSSV-004				
QC Sample	Frequency/ Number	Method/SOP — QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per preparatory batch	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL (see Box D-1 in QSM V4.2).	Correct problem; re-prepare and/or reanalyze any sample associated with a blank that fails criteria.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).
Surrogates	All field and QC samples	QC acceptance criteria specified in DoD QSM Version 4.2	Re-prepare and/or reanalyze if sufficient sample is available. If reanalysis confirms failing recoveries, report and narrate.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria specified in DoD QSM Version 4.2
LCS	One LCS per preparatory batch	QC acceptance criteria specified in DoD QSM Version 4.2	Re-prepare and/or reanalyze all associated samples.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria specified in DoD QSM Version 4.2
Internal Standards	In all field samples and standards	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard	Inspect MS or GC for malfunctions. Reanalyze all samples with internal standard. If reanalysis confirms matrix interference, report sample and narrate.	Analyst, Supervisor, QA Manager	Accuracy Bias	Retention time ± 30 seconds; EICP area within -50% to +100% of midpoint of ICAL
MS/MSD	One per preparatory batch per matrix	For matrix evaluation, use LCS recovery criteria; RPD ≤30%.	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias Precision	For matrix evaluation, use LCS recovery criteria; RPD ≤30%.

### Notes:

PAHs = Polynuclear aromatic hydrocarbons  
SIM = Selective ion monitoring  
SOP = Standard operating procedure  
GCAL = Gulf Coast Analytical  
QC = Quality assurance  
RL = Reporting limit  
QA = Quality assurance  
DoD QSM = Department of Defense Quality Systems Manual

LCS = Laboratory control sample  
ICAL = Initial calibration  
EICP = Extracted ion current profile  
MS = Mass Spectrometer  
GC = Gas chromatograph  
MS/MSD = Matrix spike/matrix spike duplicate  
RPD = Relative percent difference



## SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE (CONTINUED)

(UFP-QAPP Manual Section 3.4)

Matrix		Soil				
Analytical Group		Lead				
Analytical Method		SW-846 6020A				
SOP Reference		GCAL SOP MET-021				
QC Sample	Frequency/ Number	Method/SOP — QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per preparatory batch	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL (see Box D-1 in QSM V4.2).	Correct problem; re-prepare and/or reanalyze any sample associated with a blank that fails criteria.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).
LCS	One LCS per preparatory batch	QC acceptance criteria specified in DoD QSM Version 4.2	Re-prepare and/or reanalyze all associated samples.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria specified in DoD QSM Version 4.2
Internal Standards	In all field samples and standards	Internal standard intensity within 30-120% of intensity of the IS in the ICAL.	Reanalyze all samples with internal standard failures. If reanalysis confirms matrix interference, report sample and narrate.	Analyst, Supervisor, QA Manager	Accuracy Bias	Internal standard intensity within 30-120% of intensity of the IS in the ICAL.
Sample Duplicate or MSD	One per preparatory batch	RPD ≤ 20%	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Precision	RPD ≤ 20%
Matrix Spike	One per preparatory batch per matrix	For matrix evaluation, use LCS recovery criteria	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias	For matrix evaluation, use LCS recovery criteria
Dilution Test	One per preparatory batch	Five-fold dilution must agree within ± 10% of the original measurement for samples with concentrations > 50 x LOQ	Perform Post Digestion Spike	Analyst, Supervisor, QA Manager	Accuracy Bias	Five-fold dilution must agree within ± 10% of the original measurement for samples with concentrations > 50 times LOQ





## SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE (CONTINUED)

(UFP-QAPP Manual Section 3.4)

Matrix		Soil				
Analytical Group		Lead				
Analytical Method		SW-846 6020A				
SOP Reference		GCAL SOP MET-021				
QC Sample	Frequency/ Number	Method/SOP — QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Post Digestion Spike	When dilution test fails or analyte concentration in all samples < 50 x LOD	Recovery 75-125%	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias	Recovery 75-125%

### Notes:

SOP = Standard operating procedure  
GCAL = Gulf Coast Analytical  
QC = Quality assurance  
LOQ = Limit of quantitation  
QA = Quality assurance

DoD QSM = Department of Defense Quality Systems Manual  
LCS = Laboratory control sample  
ICAL = Initial calibration  
MSD = Matrix spike duplicate  
RPD = Relative percent difference



## SAP WORKSHEET #29: PROJECT DOCUMENTS AND RECORDS TABLE

*(UFP-QAPP Manual Section 3.5.1)*

Document	Where Maintained
<b>Field Documents</b> Field Logbook Field Sample Forms Chain of Custody Records and Air Bills Sampling Instrument Calibration Logs Sampling Notes Photographs Sampling and Analysis Plan Health and Safety Plan	Field documents will be maintained in the project file located in the Resolution Consultants Memphis, Tennessee office.
<b>Laboratory Documents</b> Sample receipt, custody, and tracking record Equipment calibration logs Sample preparation logs Analysis Run logs Corrective Action forms Reported field sample results Reported results for standards, quality control checks samples Extraction/clean-up records Raw data	Laboratory documents will be included in the hardcopy and portable documents format deliverables from the laboratory. Laboratory data deliverables will be maintained in the Resolution Consultants' Memphis, Tennessee project file and in long-term data package storage at a third-party professional document storage firm.  Electronic data results will be maintained in a database on a password protected Structured Query Language server and will be uploaded to the NIRIS database.
<b>Assessment Findings</b> Field Sampling Audit Checklist (if conducted) Analytical Audit Checklist (if conducted) Data Validation Memoranda (includes tabulated data summary forms)	All assessment documents will be maintained in the Resolution Consultants Memphis, Tennessee project file and electronically in the server library.
<b>Reports</b> Resource Conservation and Recovery Act Facility Investigation Report (final) Corrective Measures Study Report Interim Measure Work Plans Technical Memorandums Statement of Basis	All reports will be stored in hardcopy in the Resolution Consultants Memphis, Tennessee project file, electronically in the server library, and will be uploaded to the NIRIS database.



## SAP WORKSHEET #30: ANALYTICAL SERVICES TABLE

(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Sample Locations/ID Numbers	Analytical Method/SOP	Data Package Turnaround time	Laboratory/Organization <sup>1</sup> (name and address, contact person and telephone number)	Backup Laboratory
Soil & Sediment	PAHs via SIM	See Worksheet #18	SW-846 3550C/8270D EXT-001/GCMSSV-004	21 Days	GCAL, 7979 GSRI Road, Baton Rouge, LA 70820 Shelley Bourgeois, 225-214-7077	None
Soil & Sediment	Lead	See Worksheet #18	SW-846 3050B/6020A MET-004/MET-021	21 Days	GCAL, 7979 GSRI Road, Baton Rouge, LA 70820 Shelley Bourgeois, 225-214-7077	None

**Notes:**

<sup>1</sup> Laboratory meets accreditation requirements to support project needs.

PAHs = Polynuclear aromatic hydrocarbons

SIM = Selective ion monitoring

GCAL = Gulf Coast Analytical Laboratory



**SAP WORKSHEET #31: PLANNED PROJECT ASSESSMENTS TABLE**

*(UFP-QAPP Manual Section 4.1.1)*

Worksheet Not Applicable



**SAP WORKSHEET #32: ASSESSMENT FINDINGS AND CORRECTIVE ACTION RESPONSES TABLE**

*(UFP-QAPP Manual Section 4.1.2)*

Worksheet Not Applicable



## SAP WORKSHEET #33: QUALITY ASSURANCE MANAGEMENT REPORTS TABLE

(UFP QAPP Manual Section 4.2)

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Data Validation Report	Per sample delivery group	Within 4 weeks after receiving the data from the laboratory	Project Chemist or Data Validator, Resolution Consultants	TOM, Resolution Consultants; project file
Project Monthly Progress Report	Monthly for duration of the project	Monthly	TOM, Resolution Consultants	TOM, Resolution Consultants; Program Manager, Resolution Consultants; NAVFAC Midwest RPM; project file
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately upon detection of problem (on the same day)	Laboratory Project Manager, GCAL	TOM and project file, Resolution Consultants

**Notes:**

TOM = Task order manager  
RPM = Remedial project manager  
NAVFAC = Naval Facilities Engineering Command  
QA = Quality assurance  
GCAL = Gulf Coast Analytical



## SAP WORKSHEETS #34 — 36: DATA VERIFICATION AND VALIDATION (STEPS I AND IIA/IIB) PROCESS TABLE

(UFP-QAPP Manual Section 5.2.1), (UFP-QAPP Manual Section 5.2.2), (Figure 37 UFP-QAPP Manual), (Table 9 UFP-QAPP Manual)

Data Review Input	Description	Responsible for Verification (name, organization)	Step I/ IIa/IIb <sup>1</sup>	Internal/ External
<b>Verification</b> Chain-of-custody forms Sample Login/Receipt	Review the sample shipment for completeness, integrity, and sign accepting the shipment. All sample labels will be checked against the chain-of-custody form, and any discrepancies will be identified, investigated, and corrected. The samples will be logged in at every storage area and work station required by the designated analyses. Individual analysts will verify the completeness and accuracy of the data recorded on the forms.	Laboratory sample custodians and analysts, Gulf Coast Analytical Laboratories	I	Internal
<b>Verification</b> Chain-of-custody forms	Check that the chain-of-custody form was signed/dated by the sampler relinquishing the samples and by the laboratory sample custodian receiving the samples for analyses.	Project chemist or data validators, Resolution Consultants	I	External
<b>Verification</b> SAP sample tables	Verify that all proposed samples listed in the SAP tables have been collected.	FTL or designee, Resolution Consultants	I	Internal
<b>Verification</b> Sample log sheets and field notes	Verify that information recorded in the log sheets and field notes are accurate and complete.	FTL or designee, Resolution Consultants	I	Internal
<b>Verification</b> Field QC samples	Check that field QC samples, described in Worksheet #12 and listed in Worksheet #20, were collected as required.	FTL or designee, Resolution Consultants	I	Internal
<b>Verification</b> Analytical data package	Verify all analytical data packages will be verified internally for completeness by the laboratory performing the work. The laboratory project manager (or designee) will sign the case narrative for each data package.	Laboratory project manager, Gulf Coast Analytical Laboratories	I	Internal
<b>Verification</b> Analytical data package	Verify the data package for completeness. Missing information will be requested from the laboratory and validation (if performed) will be suspended until missing data are received.	FTL, Project chemist or data validators, Resolution Consultants	I	External
<b>Verification</b> Electronic data deliverables	Verify the electronic data against the chain-of-custody and hard copy data package for accuracy and completeness.	Data manager and/or validator, Resolution Consultants	I	External
<b>Validation</b> Chain-of-custody	Examine the traceability of the data from time of sample collection until reporting of data. Ensure that the custody and integrity of the samples were maintained from collection to analysis and the custody records are complete and any deviations are recorded.	Project chemist or data validators, Resolution Consultants	IIa	External
<b>Validation</b> Holding Times	Review that the samples were shipped and stored at the required temperature and sample pH for chemically-preserved samples meet the requirements listed in Worksheet #19. Ensure that the analyses were performed within the holding times. If holding times were not met, confirm that deviations were documented.	Project chemist or data validators, Resolution Consultants	IIa	External
<b>Validation</b> Sample results for representativeness	Check that the laboratory recorded the temperature at sample receipt and the pH of the chemically preserved samples to ensure sample integrity from sample collection to analysis.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External



Data Review Input	Description	Responsible for Verification (name, organization)	Step I/ IIa/IIb <sup>1</sup>	Internal/ External
<b>Validation</b> Laboratory data results for accuracy	Ensure that the laboratory QC samples were analyzed and that the measurement performance criteria, listed in Worksheet #28, were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed, as listed in Worksheet #12, and that the analytical QC criteria were met.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External
<b>Validation</b> Field and laboratory duplicate analyses for precision	Check the field sampling precision by calculating the RPD for field duplicate samples. Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses; MS/MSDs; and LCS/LCSDs. Ensure compliance with the precision goals listed in Worksheet #12 and 28.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External
<b>Validation</b> Project action limits	Assess and document the impact on matrix interferences or sample dilutions performed because of the high concentration of one or more contaminant, on the other target compounds reported as undetected.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External
<b>Validation</b> Data quality assessment report	Summarize deviations from methods, procedures, or contracts. Qualify data results based on method or QC deviation and explain all the data qualifications. Present tabular qualified data and data qualifier codes and summarize data qualification outliers. Determine if the data met the measurement performance criteria and determine the impact of any deviations on the technical usability of the data.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External
<b>Validation</b> SAP QC sample documentation	Ensure that all QC samples specified in the SAP were collected and analyzed and that the associated results were within acceptance limits.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External
<b>Validation</b> Analytical data deviations	Determine the impact of any deviation from sampling or analytical methods and laboratory SOP requirements and matrix interferences effect on the analytical results.	Project chemist or data validators, Resolution Consultants	IIb	External
<b>Validation</b> Project quantitation limits for sensitivity	Ensure that the project detection limits were achieved.	Project chemist or data validators, Resolution Consultants	IIb	External
<b>Validation</b> Sediment — PAHs via SIM	Validate PAH via SIM data using SW846 8270D method-specific criteria, data quality indicators provided in the DoD QSM, and those listed in Worksheets # 12, 19, and 28. All data will be validated and raw instrument outputs assessed and recalculated for 10% of the reported results. <i>USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review</i> , (October 1999) will be used to apply qualifiers to data to the extent possible.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External
<b>Validation</b> Sediment — Lead	Validate data using data quality indicators provided in USEPA Method 6020B method-specific criteria, data quality indicators provided in the DoD QSM, and those listed in Worksheets # 12, 19, and 28. All data will be validated and raw instrument outputs assessed and recalculated for 10% of the reported results. <i>USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review</i> , (October 2004) will be used to apply qualifiers to data to the extent possible.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External



Data Review Input	Description	Responsible for Verification (name, organization)	Step I/ IIa/IIb <sup>1</sup>	Internal/ External
Validation Data qualifiers	Qualifiers that will be applied during the data validation process are summarized below and, as indicated, results will be considered usable for interpretation unless the results are rejected when extreme data quality indicator failures are noted.	Project chemist or data validators, Resolution Consultants	IIa/IIb	External

**Notes:**

SAP = Sampling and analysis plan  
 FTL = Field team leader  
 QC = Quality control  
 RPD = Relative percent difference  
 MS/MSD = Matrix spike/Matrix Spike duplicate  
 LCS/LCSD = Laboratory control sample/laboratory control sample duplicate  
 SOP = Standard operating procedure  
 DoD QSM = *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 4.2, October 2010  
 USEPA = U.S Environmental Protection Agency  
 PAH = Polynuclear aromatic hydrocarbons  
 SIM = Selective ion monitoring

## SAP WORKSHEET #37: USABILITY ASSESSMENT

*(UFP-QAPP Manual Section 5.2.3)*

### Data Review

The usability of the data directly affects whether project objectives can be achieved and the following characteristics will be evaluated at a minimum. The results of these evaluations will be included in the project report. The characteristics will be evaluated for multiple concentration levels if the evaluator determines that this is necessary. To the extent required by the type of data being reviewed, the assessors will consult with other technically competent individuals to render sound technical assessments of these data characteristics:

- **Completeness** — Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount expected to be obtained under correct normal conditions. It is expected that 100% of the planned sampling points will be collected. The completeness goal for field measurements will be greater than 90%. Laboratory analysis for this project will have a completeness goal greater than 95% to account for unanticipated results that may be rejected during data validation. Completeness can be calculated using the following equation.

$$\%Completeness = \frac{No. of Valid Tests}{Total Tests Taken} \times 100$$

The FTL, acting on behalf of the Project Team, will determine whether deviations from the scheduled sample collection or analyses occurred. If they have occurred and the Resolution Consultants TOM determines that the deviations compromise the ability to meet project objectives he will consult with the Navy RPM and other project team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions.

- **Precision** — Precision measures the reproducibility of measurements and methods and is defined for qualitative data as a group of values' variability compared with its average value. To assess the precision of the measurement systems used in this project, field duplicates will be obtained and analyzed with the samples collected. Precision of laboratory analysis will be assessed by comparing the relative percent difference (RPD) of analytical results between MS and MSDs (or sample duplicates) and the measurement quality objectives will be those cited in Worksheets #12 and #28. The RPD will be calculated for each pair of duplicate analysis using the following equation:

$$RPD = \frac{(S - D)}{(S + D) / 2} \times 100$$

Where:

S = sample result  
D = duplicate result

The project chemist, acting on behalf of the Project Team, will determine whether precision goals for field duplicates and laboratory duplicates were met. This will be accomplished by comparing duplicate results to precision goals identified in Worksheets #12 and #28. This also will include a comparison of field and laboratory precision with the expectation that laboratory duplicate results will be no less precise than field duplicate results. If the goals are not met or data have been flagged as estimated (J qualifier), limitations on the use of the data will be described in the project report.

- **Accuracy** — Accuracy is the degree to which a given result agrees with the true value. The accuracy of an entire measurement system is an indication of any bias that exists. Spiked sample results provide information needed to assess the accuracy of analyses. Specifically, surrogate spike, MS/MSD, and laboratory control sample (LCS) percent recoveries (%Rs) are used to assess accuracy. Every organic sample is spiked with known quantities of non-target surrogate compounds. Five percent of all samples analyzed are spiked with target chemicals for the MS/MSD (or sample duplicates). If the calculated %Rs for the known spike concentrations are within defined control limits set by each method, the reported sample concentrations are considered accurate. The accuracy measurement quality objectives will be those cited in Worksheets #12 and #28. Accuracy is calculated using the following equation:

$$\% R = \frac{(SSR - SR)}{SA} \times 100$$

Where:

SSR = spike sample recovery  
SR = sample recovery  
SA = concentration of spike added

The project chemist, acting on behalf of the Project Team, will determine whether the accuracy/bias goals were met for project data. This assessment will include an evaluation of field and laboratory contamination; instrument calibration variability; and analyte recoveries for surrogates, matrix spike, matrix spike duplicate, and laboratory control

samples against the goals identified in worksheets #24 and #28. If the goals are not met, limitations on the use of the data will be described in the project report. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report.

- **Representativeness** — A project scientist, identified by the Resolution Consultants TOM and acting on behalf of the Project Team, will determine whether the data are adequately representative of intended populations, both spatially and temporally. This will be accomplished by verifying that samples were collected and analyzed in accordance with this SAP, by reviewing spatial and temporal data variations, and by comparing these characteristics to expectations. The usability report will describe the representativeness of the data for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the project scientist indicates that a quantitative analysis is required.
- **Comparability** — The project chemist, acting on behalf of the Project Team, will determine whether the data generated under this project are sufficiently comparable to historical property data generated by different methods and for samples collected using different procedures and under different property conditions. This will be accomplished by comparing overall precision and bias among data sets for each matrix and analytical fraction. This will not require quantitative comparisons unless the project chemist indicates that such quantitative analysis is required.
- **Sensitivity** — The project chemist, acting on behalf of the Project Team, will determine whether project sensitivity goals listed in Worksheet #15 are achieved. The overall sensitivity and quantitation limits from multiple data sets for each matrix and analysis will be compared. If sensitivity goals are not achieved, the limitations on the data will be described.

**Describe the evaluative procedures used to assess overall measurement error associated with the project:**

After completion of the data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. In addition to the evaluations described above, a series of inspections and statistical analyses will be performed to estimate these characteristics. The statistical evaluations will include simple summary statistics for



target analytes, such as maximum concentration, minimum concentration, number of samples exhibiting non-detected results, number of samples exhibiting positive results, and the proportion of samples with detected and non-detected results. The Project Team members, identified by the Resolution Consultants TOM, will assess whether the data collectively support the attainment of project objectives. They will consider whether any missing or rejected data have compromised the ability to make decisions or to make the decisions with the desired level of confidence. The data will be evaluated to determine whether missing or rejected data can be compensated by other data. Although rejected data will generally not be used, there may be reason to use them in a weight-of-evidence argument, especially when they supplement data that have not been rejected. If rejected data are used, their use will be supported by technically defensible rationales.

**Identify the personnel responsible for performing the usability assessment:**

The Resolution Consultants TOM, project chemist, and FTL will be responsible for conducting the listed data usability assessments. The data usability assessment will be reviewed with the Project Team. If deficiencies affecting the attainment of project objectives are identified, the review will take place either in a face to face meeting or a teleconference depending on the extent of identified deficiencies. If no significant deficiencies are identified, the data usability assessment will simply be documented in the project report and reviewed during the normal document review cycle.

**Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:**

The data will be presented in tabular format, including data qualifications such as estimation (J, UJ) or rejection (R). The project report will identify and describe the data usability limitations and suggest re-sampling or other corrective actions, if necessary. Graphical presentations of the data such as concentration tag maps will be generated as part of the overall data evaluation process.



## REFERENCES

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**Appendix A**  
**Standard Operating Procedures**

# Utility Clearance

## Procedure 3-01

### 1.0 Purpose and Scope

- 1.1 This standard operating procedure (SOP) describes the process for determining the presence of subsurface utilities and other cultural features at locations where planned site activities involve the physical disturbance of subsurface materials.
- 1.2 This procedure is the Program-approved professional guidance for work performed by Resolution Consultants under the Comprehensive Long-Term Environmental Action Navy (CLEAN) contract (Contract Number N62470-11-D-8013).
- 1.3 The procedure applies to the following activities: soil gas surveying, excavating, trenching, drilling of borings and installation of monitoring and extraction wells, use of soil recovery or slide-hammer hand augers, and all other intrusive sampling activities.
- 1.4 The primary purpose of the procedure is to minimize the potential for damage to underground utilities and other subsurface features, which could result in physical injury, disruption of utility service, or disturbance of other subsurface cultural features.
- 1.5 If there are procedures, whether it be from Resolution Consultants, state, and/or federal, that are not addressed in this SOP and are applicable to utility clearance, those procedures should be added as an appendix to the project specific SAP.
- 1.6 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

### 2.0 Safety

- 2.1 Field and subcontractor personnel shall adhere to a site-specific health and safety plan (HASP).

### 3.0 Terms and Definitions

#### 3.1 Utility

For the purposes of this SOP, a utility is defined as a manmade underground line or conduit, cable, pipe, vault or tank that is, or was, used for the transmission of material or energy (e.g., gas, electrical, telephone, steam, water or sewage, product transfer lines, or underground storage tanks).

#### 3.2 As-Built Plans

As-built plans are plans or blueprints depicting the locations of structures and associated utilities on a property.

#### 3.3 One-Call

The Utility Notification Center is the one-call agency for nationwide call before you dig. The Utility Notification Center is open 24 hours a day, and accepts calls from anyone planning to dig. The phone number 811 is the designated call before you dig phone number that directly connects you to your local one-call center. Additional information can be found at [www.call811.com](http://www.call811.com).

Calling before you dig ensures that any publicly owned underground lines will be marked so that you can dig around them safely. Having the utility lines marked not only prevents accidental damage to the lines, but prevents property damage and personal injuries that could result in breaking a line.

The following information will need to be provided when a call is placed to One-Call:

- Your name, phone number, company name (if applicable), and mailing address.
- What type of work is being done.
- Who the work is being done for.
- The county and city the work is taking place in.
- The address or the street where the work is taking place.
- Marking instructions, (specific instructions as to where the work is taking place).

Under normal circumstances it takes between 2 to 5 days from the time you call (not counting weekends or holidays) to have the underground lines marked. Because these laws vary from state to state, exactly how long it will take depends on where your worksite is located. You will be given an exact start time and date when your locate request is completed, which will comply with the laws in your area.

In the event of an emergency (any situation causing damage to life or property, or a service outage), lines can be marked sooner than the original given time if requested.

#### 3.4 Toning

Toning is the process of surveying an area utilizing one or more surface geophysical methods to determine the presence or absence of underground utilities. Typically, toning is conducted after identifying the general location of utilities and carefully examining all available site utility plans. Each location is marked according to the type of utility being identified. In addition, areas cleared by toning are flagged or staked to indicate that all identified utilities in a given area have been toned.

### 4.0 Training and Qualifications

- 4.1 The **Contract Task Order (CTO) Manager** is responsible for verifying that these utility locating procedures are performed prior to the initiation of active subsurface exploration.
- 4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.
- 4.3 The **Field Manager** is responsible for ensuring that all utility locating activities are performed in accordance with this procedure.
- 4.4 All **Field Personnel** are responsible for the implementation of this procedure.

### 5.0 Equipment and Supplies

- 5.1 Equipment and supplies necessary for locating subsurface utilities will be provided by the subcontractor; however, the project **Field Manager/Field Personnel** will provide any additional equipment and supplies as needed as well as maintain information regarding the utility clearance activities in the field logbook.

### 6.0 Procedure

Proceed with the following steps where subsurface exploration will include excavations, drilling, or any other subsurface investigative method that could damage utilities at a site. In addition to the steps outlined below, always exercise caution while conducting subsurface exploratory work.



#### 6.1 **Prepare Preliminary Site Plan**

- Prepare a preliminary, scaled site plan depicting the proposed exploratory locations as part of the project specific Sampling and Analysis Plan (SAP) or Work Plan. Include as many of the cultural and natural features as practical in this plan.

#### 6.2 **Review Background Information**

- Search existing plan files to review the as-built plans to identify the known location of utilities at the site. Plot the locations of utilities identified onto a preliminary, scaled site plan. Inform the CTO Manager if utilities lie within close proximity to a proposed exploration or excavation location. The CTO Manager will determine if it is necessary to relocate proposed sampling or excavation locations.
- Include the utility location information gathered during previous investigations (e.g., remedial investigation or remedial site evaluation) in the project design documents for removal or remedial actions. In this manner, information regarding utility locations collected during implementation of a CTO can be shared with the subcontractor during implementation of a particular task order. In many instances, this will help to reduce the amount of additional geophysical surveying work the subcontractor may have to perform.
- Conduct interviews with onsite and facility personnel familiar with the site to obtain additional information regarding the known and suspected locations of underground utilities. In addition, if appropriate, contact shall be made with local utility companies to request their help in locating underground lines. Pencil in the dimensions, orientation, and depth of utilities, other than those identified on the as-built plans, at their approximate locations on the preliminary plans. Enter the type of utility, the personnel who provided the information, and the date the information was provided into the field log.
- During the pre-field work interviewing process, the interviewer will determine which site personnel should be notified in the event of an incident involving damage to existing utilities. Record this information in the field logbook with the corresponding telephone numbers and addresses.

#### 6.3 **Site Visit/Locate Utilities/Toning**

- Prior to the initiation of field activities, the Field Task Manager or similarly qualified field personnel shall visit the site and note existing structures and evidence of associated utilities, such as fire hydrants, irrigation systems, manhole and vault box covers, standpipes, telephone switch boxes, free-standing light poles, gas or electric meters, pavement cuts, and linear depression. Compare notes of the actual site configuration to the preliminary site plan. Note deviations in the field logbook and on the preliminary site plan. Accurately locate or survey and clearly mark with stakes, pins, flags, paint, or other suitable devices all areas where subsurface exploration is proposed. These areas shall correspond with the locations drawn on the preliminary site plan.
- Following the initial site visit by the Field Task Manager, a trained utility locating subcontractor will locate, identify, and tone all utilities depicted on the preliminary site plan. The Field Task Manager or similarly qualified field personnel shall visit the site and identify the areas of subsurface disturbance with white spray paint, chalk, white pin flags or some other easily identifiable marking. The utility locator should utilize appropriate sensing equipment to attempt to locate utilities that might not have appeared on the as-built plans. At a minimum, the utility subcontractor should utilize a metal detector and/or magnetometer; however, it is important to consider the possibility that non-metallic utilities or tanks might be present at the site. Use other appropriate surface geophysical methods such as Ground Penetrating Radar, Radiodetection, etc. as appropriate. Clear proposed exploration areas of all utilities in the immediate area where subsurface exploration is proposed. Clearly tone all anomalous areas. Clearly identify all toned areas on the preliminary site plan. All utilities near the area of subsurface disturbance should also be marked out by the utility subcontractor using the universal colors for subsurface utilities (i.e., red – electric; blue – water; green – sewer; yellow – gas; etc.). After toning the site and plotting all known or suspected buried utilities on the preliminary site plan, the utility locator shall provide the Field Task Manager with a copy of the completed preliminary

site plan. Alternatively, the Field Task Manager or designee shall document the results of the survey on the preliminary site plan.

- Report to the Field Task Manager anomalous areas detected and toned that are in close proximity to the exploration or excavation areas. The Field Task Manager shall determine the safe distance to maintain from the known or suspected utility. It may be necessary to relocate the proposed exploration or excavation areas. If this is required, the Field Task Manager or designee shall relocate them and clearly mark them using the methods described above. Completely remove the markings at the prior location. Plot the new locations on the site plan and delete the prior locations from the plan. In some instances, such as in areas extremely congested with subsurface utilities, it may be necessary to dig by hand or use techniques such as air knife to determine the location of the utilities.

#### 6.4 **Prepare Site Plan**

- Prior to the initiation of field activities, draft a final site plan that indicates the location of subsurface exploration areas and all known or suspected utilities present at the site. Provide copies of this site plan to the Navy Technical Representative (NTR), the CTO Manager, and the subcontractor who is to conduct the subsurface exploration/excavation work. Review the site plan with the NTR to verify its accuracy prior to initiating subsurface sampling activities.

### 7.0 **Quality Control and Assurance**

- 7.1 Utility locating must incorporate quality control measures to ensure conformance to these and the project requirements.

### 8.0 **Records, Data Analysis, Calculations**

- 8.1 A bound field logbook will be kept detailing all activities conducted during the utility locating procedure.
- 8.2 The logbook will describe any changes and modifications made to the original exploration plan. The trained utility locator shall prepare a report and keep it in the project file. Also, a copy of the final site plan will be kept in the project file.

### 9.0 **Attachments or References**

Department of Defense, United States (DoD). 2005. [Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual](http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf). Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: [http://www.epa.gov/fedfac/pdf/ufp\\_qapp\\_v1\\_0305.pdf](http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf).

Author	Reviewer	Revisions (Technical or Editorial)
Caryn DeJesus Senior Scientist	Bob Shoemaker Senior Scientist	Rev 0 – Initial Issue (June 2012)

# Logbooks

## Procedure 3-02

### 1.0 Purpose and Scope

- 1.1 This standard operating procedure (SOP) describes the activities and responsibilities pertaining to the identification, use, and control of logbooks and associated field data records.
- 1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

### 2.0 Safety

- 2.1 In order to keep the logbook clean, store it in a clean location and use it only when outer gloves used for PPE have been removed.

### 3.0 Terms and Definitions

#### 3.1 Logbook

A logbook is a bound field notebook with consecutively numbered, water-repellent pages that is clearly identified with the name of the relevant activity, the person assigned responsibility for maintenance of the logbook, and the beginning and ending dates of the entries.

#### 3.2 Data Form

A data form is a predetermined format utilized for recording field data that may become, by reference, a part of the logbook (e.g., soil boring logs, trenching logs, surface soil sampling logs, groundwater sample logs, and well construction logs are data forms).

### 4.0 Training and Qualifications

- 4.1 The **Contract Task Order (CTO) Manager** or **designee** is responsible for determining which team members shall record information in field logbooks and for obtaining and maintaining control of the required logbooks. The **CTO Manager** shall review the field logbook on at least a monthly basis. The **CTO Manager** or **designee** is responsible for reviewing logbook entries to determine compliance with this procedure and to ensure that the entries meet the project requirements.
- 4.2 A knowledgeable individual such as the **Field Manager, CTO Manager, or Program Quality Manager** shall perform a technical review of each logbook at a frequency commensurate with the level of activity (weekly is suggested, or, at a minimum, monthly). Document these reviews by the dated signature of the reviewer on the last page or page immediately following the material reviewed.
- 4.3 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.
- 4.4 The **Field Manager** is responsible for ensuring that all **field personnel** follow these procedures and that the logbook is completed properly and daily. The **Field Manager** is also responsible for submitting copies to the **CTO Manager**, who is responsible for filing them and submitting a copy (if required by the CTO Statement of Work).
- 4.5 The **logbook user** is responsible for recording pertinent data into the logbook to satisfy project requirements and for attesting to the accuracy of the entries by dated signature. The **logbook user** is also responsible for safeguarding the logbook while having custody of it.

4.6 All **field personnel** are responsible for the implementation of this procedure.

## **5.0 Equipment and Supplies**

5.1 Field logbooks shall be bound field notebooks with water-repellent pages.

5.2 Pens shall have indelible black ink.

## **6.0 Procedure**

6.1 The field logbook serves as the primary record of field activities. Make entries chronologically and in sufficient detail to allow the writer or a knowledgeable reviewer to reconstruct the applicable events. Store the logbook in a clean location and use it only when outer gloves used for personal protective equipment (PPE) have been removed.

6.2 Individual data forms may be generated to provide systematic data collection documentation. Entries on these forms shall meet the same requirements as entries in the logbook and shall be referenced in the applicable logbook entry. Individual data forms shall reference the applicable logbook and page number. At a minimum, include names of all samples collected in the logbook even if they are recorded elsewhere.

6.3 Enter field descriptions and observations into the logbook, as described in Attachment 1, using indelible black ink.

6.4 Typical information to be entered includes the following:

- Dates (month/day/year) and times (military) of all on-site activities and entries made in logbooks/forms;
- Site name and description;
- Site location by longitude and latitude, if known;
- Weather conditions, including temperature and relative humidity;
- Fieldwork documentation, including site entry and exit times;
- Descriptions of, and rationale for, approved deviations from the work plan (WP) or field sampling plan;
- Field instrumentation readings;
- Names, job functions, and organizational affiliations of on-site personnel;
- Photograph references;
- Site sketches and diagrams made on site;
- Identification and description of sample morphology, collection locations, and sample numbers;
- Sample collection information, including dates (month/day/year) and times (military) of sample collections, sample collection methods and devices, station location numbers, sample collection depths/heights, sample preservation information, sample pH (if applicable), analysis requested (analytical groups), etc., as well as chain-of-custody (COC) information such as sample identification numbers cross-referenced to COC sample numbers;
- Sample naming convention;
- Field quality control (QC) sample information;
- Site observations, field descriptions, equipment used, and field activities accomplished to reconstruct field operations;

- Meeting information;
  - Important times and dates of telephone conversations, correspondence, or deliverables;
  - Field calculations;
  - PPE level;
  - Calibration records;
  - Contractor and subcontractor information (address, names of personnel, job functions, organizational affiliations, contract number, contract name, and work assignment number);
  - Equipment decontamination procedures and effectiveness;
  - Laboratories receiving samples and shipping information, such as carrier, shipment time, number of sample containers shipped, and analyses requested; and
  - User signatures.
- 6.5 The logbook shall reference data maintained in other logs, forms, etc. Correct entry errors by drawing a single line through the incorrect entry, then initialing and dating this change. Enter an explanation for the correction if the correction is more than for a mistake.
- 6.6 At least at the end of each day, the person making the entry shall sign or initial each entry or group of entries.
- 6.7 Enter logbook page numbers on each page to facilitate identification of photocopies.
- 6.8 If a person's initials are used for identification, or if uncommon acronyms are used, identify these on a page at the beginning of the logbook.
- 6.9 At least weekly and preferably daily, the **preparer** shall photocopy and retain the pages completed during that session for backup. This will prevent loss of a large amount of information if the logbook is lost.

## **7.0 Quality Control and Assurance**

- 7.1 Review per Section 4.2 shall be recorded.

## **8.0 Records, Data Analysis, Calculations**

- 8.1 Retain the field logbook as a permanent project record. If a particular CTO requires submittal of photocopies of logbooks, perform this as required.
- 8.2 Deviations from this procedure shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

## **9.0 Attachments or References**

- 9.1 Attachment 1 – Description of Logbook Entries
- 9.2 Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: [http://www.epa.gov/fedfac/pdf/ufp\\_qapp\\_v1\\_0305.pdf](http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf).

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue

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## Attachment 1

### Description of Logbook Entries

Logbook entries shall be consistent with Section A.1.4 *Field Documentation SOPs* of the UFP-QAPP Manual (DoD 2005) and contain the following information, as applicable, for each activity recorded. Some of these details may be entered on data forms, as described previously.

Name of Activity	For example, Asbestos Bulk Sampling, Charcoal Canister Sampling, Aquifer Testing.
Task Team Members and Equipment	Name all members on the field team involved in the specified activity. List equipment used by serial number or other unique identification, including calibration information.
Activity Location	Indicate location of sampling area as indicated in the field sampling plan.
Weather	Indicate general weather and precipitation conditions.
Level of PPE	Record the level of PPE (e.g., Level D).
Methods	Indicate method or procedure number employed for the activity.
Sample Numbers	Indicate the unique numbers associated with the physical samples. Identify QC samples.
Sample Type and Volume	Indicate the medium, container type, preservative, and the volume for each sample.
Time and Date	Record the time and date when the activity was performed (e.g., 0830/08/OCT/89). Use the 24-hour clock for recording the time and two digits for recording the day of the month and the year.
Analyses	Indicate the appropriate code for analyses to be performed on each sample, as specified in the WP.
Field Measurements	Indicate measurements and field instrument readings taken during the activity.
Chain of Custody and Distribution	Indicate chain-of-custody for each sample collected and indicate to whom the samples are transferred and the destination.
References	If appropriate, indicate references to other logs or forms, drawings, or photographs employed in the activity.
Narrative (including time and location)	<p>Create a factual, chronological record of the team's activities throughout the day including the time and location of each activity. Include descriptions of general problems encountered and their resolution. Provide the names and affiliations of non-field team personnel who visit the site, request changes in activity, impact the work schedule, request information, or observe team activities. Record any visual or other observations relevant to the activity, the contamination source, or the sample itself.</p> <p>It should be emphasized that logbook entries are for recording data and chronologies of events. The logbook author must include observations and descriptive notations, taking care to be objective and recording no opinions or subjective comments unless appropriate.</p>
Recorded by	Include the signature of the individual responsible for the entries contained in the logbook and referenced forms.
Checked by	Include the signature of the individual who performs the review of the completed entries.

# Sample Labeling and Chain of Custody Procedures

## Procedure 3-03 (MS)

### 1.0 Purpose and Scope

- 1.1 The purpose of this standard operating procedure is to establish standard protocols for all field personnel for use in maintaining field and sampling activity records, labeling samples, ensuring that proper sample custody procedures are utilized, and completing chain-of-custody/analytical request forms.
- 1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

### 2.0 Safety

Not applicable

### 3.0 Definitions

#### 3.1 Logbook

A logbook is a bound field notebook with consecutively numbered, water-repellent pages that is clearly identified with the name of the relevant activity, the person responsible for maintenance of the logbook, and the beginning and ending dates of the entries.

#### 3.2 Chain-of-Custody

Chain-of-custody (COC) is documentation of the process of custody control. Custody control includes possession of a sample from the time of its collection in the field to its receipt by the analytical laboratory, and through analysis and storage prior to disposal.

### 4.0 Training and Qualifications

- 4.1 The **CTO Manager**, or designee, is responsible for determining which team members shall record information in the field logbook and for checking sample logbooks and COC forms to ensure compliance with these procedures. The **CTO Manager**, or designee, shall review COC forms at the completion of each sampling event.
- 4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.
- 4.3 The **Field Manager** is responsible for ensuring that all field equipment is decontaminated according to this procedure.
- 4.4 The **Project Chemist**, or designee, is responsible for verifying that the COC/analytical request forms have been completed properly and match the sampling and analytical plan. The **Project Chemist**, or designee, is responsible for notifying the laboratory, data managers, and data validators in writing if analytical request changes are required as a corrective action. These small changes are different from change orders, which involve changes to the scope of the subcontract with the laboratory and must be made in accordance with a respective contract.
- 4.5 All **Field Personnel** are responsible for recording pertinent data onto the COC forms to satisfy project requirements and for attesting to the accuracy of the entries by dated signature.

## 5.0 Procedure

This procedure provides standards for labeling the samples, documenting sample custody, and completing COC/analytical request forms. The standards presented in this section shall be followed to ensure that samples collected are maintained for their intended purpose and that the conditions encountered during field activities are documented.

### 5.1 Sample Labeling

Affix a waterproof sample label with adhesive backing to each individual sample container. Record the following information with a waterproof marker on each label:

- Project name or number (optional)
- COC sample number
- Date and time of collection
- Sampler's initials
- Matrix (optional)
- Sample preservatives (if applicable)
- Analysis to be performed on sample (This shall be identified by the method number or name identified in the subcontract with the laboratory)

These labels may be obtained from the analytical laboratory or printed from a computer file onto adhesive labels.

### 5.2 Custody Procedures

For samples intended for chemical analysis, sample custody procedures shall be followed through collection, transfer, analysis, and disposal to ensure that the integrity of the samples is maintained. A description of sample custody procedures is provided below.

#### Sample Collection Custody Procedures

According to the EPA guidelines, a sample is considered to be in custody if one of the following conditions is met:

- It is in one's actual physical possession or view
- It is in one's physical possession and has not been tampered with (i.e., it is under lock or official seal)
- It is retained in a secured area with restricted access
- It is placed in a container and secured with an official seal such that the sample cannot be reached without breaking the seal

Place custody seals on shipping coolers (and sample jars, if required) if the cooler/container is to be removed from the sampler's custody. Place a minimum of two custody seals in such a manner that they must be broken to open the containers or coolers. Label the custody seals with the following information:

- Sampler's name or initials
- Date and time that the sample/cooler was sealed

These seals are designed to enable detection of sample tampering. An example of a custody seal is shown in Attachment 1.

Field personnel shall also log individual samples onto COC forms (carbon copy or computer generated) when a sample is collected. These forms may also serve as the request for analyses. Procedures for completing these forms are discussed in Section 0, indicating sample identification number, matrix, date and time of collection, number of containers, analytical methods to be performed on the sample, and preservatives added (if any). The samplers will also sign the COC form signifying that they were the personnel who collected the samples. The COC form shall accompany the samples from the field to the laboratory. When a cooler is ready for shipment to the analytical laboratory, the person delivering the samples for transport will sign and indicate the date and time on the accompanying COC form. One copy of the COC form will be retained by the sampler and the remaining copies of the COC form shall be placed inside a self-sealing bag and taped to the inside of the cooler. Each cooler must be associated with a unique COC form. Whenever a transfer of custody takes place, both parties shall sign and date the accompanying carbon copy COC forms, and the individual relinquishing the samples shall retain a copy of each form. One exception is when the samples are shipped; the delivery service personnel will not sign or receive a copy because they do not open the coolers. The laboratory shall attach copies of the completed COC forms to the reports containing the results of the analytical tests. An example COC form is provided in Attachment 2.

### 5.3 Completing COC/Analytical Request Forms

COC form/analytical request form completion procedures are crucial in properly transferring the custody and responsibility of samples from field personnel to the laboratory. This form is important for accurately and concisely requesting analyses for each sample; it is essentially a release order from the analysis subcontract.

Attachment 2 is an example of a completed COC/analytical request form that may be used by field personnel, with box numbers identified and discussed in text below. Multiple copies may be tailored to each project so that much of the information described below need not be handwritten each time. Each record on the form (Attachment 2) is identified with a bold number corresponding to the instructions given below.

1. Record the project name, site location.
2. Record the site location, including the state.
3. Record the Contract Task Order number
4. Record the Resolution Consultants Task Order Manager
5. Record the sampler/site phone or cell number (if applicable).
6. Record the laboratory name where the samples were sent.
7. Record the requested turnaround time, in days. If a specific turnaround time is required to meet project objectives, but was not indicated on the laboratory service request form submitted to the purchasing department, the sampler, project manager, or site manager should contact the purchasing department so the laboratory contract can be modified.
8. Record the COC number that is defined by the sampler and should be unique throughout the project's history. An example would be to use the sampler's initials followed by the data. If multiple custodies are generated on a given day, use a unique sequential identifier. Example: CRC040105A, CRC040105B
9. Record the purchase order number provided by the purchasing department.
10. Record the page and total number of COC forms used in a shipment.
11. Record the project, and phase applicable to the sampling task.
12. Record the two-character code corresponding to the *chemical* preservation type, which is found on the bottom of the COC form. If no chemical preservation was added to the sample, the field should be left blank. Temperature preservation need not be documented at this location, but will be indicated elsewhere on the COC form (see 33).

13. List the requested analysis. Whenever possible, list the corresponding analytical method. (e.g., VOCs, 8260).
14. For Lab identification use only.
15. Record the full unique sample identification as detailed in the Site's Sampling and Analysis Plan.
16. Record the location identification, which is a shortened ID used for presentation and mapping, as detailed in the Site's Sampling and Analysis Plan.
17. Record the sample date using the format mm/dd/yy.
18. Record the sample time using the military format of hhmm.
19. Record the matrix code of the sample, which is located at the bottom of the COC form. The matrix code is a crucial element of the Navy's data management system. For simplicity, only typical matrix codes are listed on the bottom COC form, but below is a complete listing of all applicable Navy matrix codes:

**Table 1  
Navy Matrix Codes**

Matrix Code	Matrix Code Description	Matrix Code	Matrix Code Description
AA	Ambient air	RK	Rock
AC	Composite air sample	SB	Bentonite
AD	Air - Drilling	SBS	Sub-surface soil ( > 6")
AIN	Integrated air sample (under sample form of gas)	SC	Cement/Concrete
AQ	Air quality control matrix	SD	Drill cuttings — solid matrix
AQS	Aqueous	SE	Sediment
ASB	Asbestos	SEEP	SEEP
ASBF	Asbestos-Fibrous	SF	Filter sand pack
ASBNF	Asbestos-Non-Fibrous	SJ	Sand
AVE	Air-Vapor extraction, effluent	SK	Asphalt
AX	Air sample from unknown origin	SL	Sludge
BK	Brick	SM	Water filter (solid material used to filter water)
BS	Brackish sediment	SN	Miscellaneous solid/building materials
CA	Cinder ash	SO	Soil
CK	Caulk	SP	Casing (PVC, stainless steel, cast iron, iron pipe)
CN	Container	SQ	Soil/Solid quality control matrix
CR	Carbon (usually for a remediation system)	SS	Scrapings
DF	Dust/Fallout	SSD	Subsurface sediment
DR	Debris/rubble	STKG	Stack gas
DS	Storm drain sediment	STPM	Stripper Tower Packing Media
DT	Trapped debris	SU	Surface soil (less than 6 inches)
EF	Emissions flux	SW	Swab or wipe
EW	Elutriate water	SZ	Wood
FB	Fibers	TA	Animal tissue
FL	Forest litter	TP	Plant tissue
GE	Soil gas effluent — stack gas (from system)	TQ	Tissue QC
GI	Soil gas influent (into system)	TX	Tissue
GL	Headspace of liquid sample	UNK	Unknown
GQ	Gaseous or Headspace QC	W	Water (not groundwater, unspecified)
GR	Gravel	WA	Drill cuttings - aqueous mix
GS	Soil gas	WB	Brackish Water

**Table 1**  
**Navy Matrix Codes**

<b>Matrix Code</b>	<b>Matrix Code Description</b>	<b>Matrix Code</b>	<b>Matrix Code Description</b>
GT	Grit	WC	Drilling water (used for well construction)
IC	IDW Concrete	WD	Well development water
IDD	IDW Solid	WF	Freshwater (not groundwater)
IDS	IDW soil	WG	Ground water
IDW	IDW Water	WH	Equipment wash water
IW	Interstitial water	WI	Ground water influent (into system)
LA	Aqueous phase of a multiphase liquid/soil	WL	Leachate
LF	Product (floating or free)	WM	Marine water
LQ	Organic liquid quality control matrix	WN	Pore water
MA	Mastic	WO	Ocean water
MO	Mortar	WP	Drinking water
MR	Marine sediment	WQ	Water for QC samples
MS	Metal shavings	WR	Ground water effluent (from system)
NS	Near-surface soil	WS	Surface water
PA	Paper	WT	Composite groundwater sample
PC	Paint Chips	WU	Storm water
PP	Precipitate	WW	Waste water
RE	Residue		

**Field QC blanks** will require matrix codes that identify the type of blank associated with parent sample. Aqueous field QC blanks are not automatically identified with a matrix code of "WQ," indicating a water quality control blank; they are only identified with a matrix code of "WQ" if the associated samples are also aqueous. Trip blanks, field blanks, and equipment rinsate blanks collected in association with **soil** samples will be identified with a matrix code of "SQ," even though the actual matrix is aqueous, because the blanks were collected to assess potential contamination imparted during decontamination activities or transport of **soil** samples.

20. Record the sample type code, which is located at the bottom of the COC form. The sample type is a crucial element of the EQuIS data management system. For simplicity, only typical sample type codes are listed on the bottom of the COC form, but below is a list of all applicable Navy field sample type codes:

**Table 2**  
**Navy Sample Type Codes**

<b>Sample Type Code</b>	<b>Sample Type Code Description</b>
AB	Ambient condition blank
BIOCON	Bioassay control sample
BS	Blank spike
BSD	Blank spike duplicate
EB	Equipment blank
EBD	Equipment blank/rinsate duplicate
FB	Field blank
FD	Field duplicate
FS	Field spike
IDW	Purge and rinsate water
LB	Lab Blank

**Table 2**  
**Navy Sample Type Codes**

<b>Sample Type Code</b>	<b>Sample Type Code Description</b>
LR	Lab Replicate
MB	Material blank
MIS	Multi-Incremental Sample
MS	Matrix spike
N	Normal (Regular)
PE	Performance evaluation
PURGE	Purge water sample
RD	Regulatory duplicate
SB	Source blank
SBD	Source blank duplicate
SCREEN	Screening Sample
SD	Matrix spike duplicate
SPLIT	Sample split
SRM	Standard reference material
TB	Trip Blank
TBD	Trip blank duplicate
TBR	Trip blank replicate

Field duplicate samples — Field duplicates will be identified using the format detailed in the Site's Sampling and Analysis Plan. However, field duplicates will also be differentiated from the parent sample on the chain-of-custody form. The parent sample will have a sample type code of "N," for normal environmental sample; while its duplicate will have a sample type code of "FD."

21. Record whether the sample is field filtered with a "Y" or not field filtered with an "N." If a project requires collecting samples for both total and dissolved constituents, the same sample and location ID is used for both (see 15 and 16); however, the sampler will indicate whether the sample is field filtered at this location on the COC form. This field must always be filled out; even when soil samples are collected (where "N" appropriately applies, in most cases).
22. Record the total number of containers that are submitted for all of the tests. This must add up to the total number of containers listed for each individual test in 23.
23. Record the number of containers for each test. Do not use Xs, rather indicate the number of containers submitted for each test listed in 14. For example, Sample 010MW007002 requires analysis for VOCs (8260), and SVOCs (8270). Record 3 under the VOC analysis and 2 under the SVOC (assuming 3 containers were submitted for VOCs and 2 were submitted for SVOCs). The total number of containers in this example is 5, which should be the total number of containers listed in 22. Extra containers submitted for matrix spike/matrix spike duplicates (MS/MSDs) will be appropriately recorded.
24. Indicate if extra sample volume was included for MS/MSD analysis using an "X." Samples to be used for MS/MSDs will use the same sample ID and location ID (see 15 and 16), but will be collected in triplicate, particularly for liquid samples, to ensure the analytical laboratory receives sufficient volume for the analyses.
25. Indicate if the samples should be held by the laboratory for future testing using an "X."
26. Record any field comments.
27. Reserved for laboratory comments.



28. Indicate the total number of coolers in each shipment. *Note:* When multiple coolers are submitted, each should contain a COC form.
29. Signature(s) of the person(s) relinquishing sample custody.
30. Signature(s) of the person(s) receiving sample custody.
31. Indicate whether the samples are iced, by checking the appropriate response.
32. Indicate the method of shipment (e.g., FedEx, hand-delivered, laboratory courier).
33. Record the airbill number when a commercial courier is used. This is particularly important when multiple coolers are sent in the same shipment or when the laboratory is sent the COC form in advance of receiving samples because it aids in tracking lost coolers.
34. Record the date the coolers were shipped.

COC forms tailored to each CTO can be drafted and printed onto multiple forms. This eliminates the need to rewrite the analytical methods column headers each time. It also eliminates the need to write the project manager, name, and number; QC Level; turnaround time; and the same general comments each time.

Complete one COC form per cooler. Whenever possible, place all volatile organic analyte vials into one cooler in order to reduce the number of trip blanks. Complete all sections and be sure to sign and date the COC form. One copy of the COC form must remain with the field personnel.

## 6.0 Records

The COC/analytical request form shall be faxed or emailed approximately daily to the Project Chemist, or designee for verification of accuracy. Following the completion of sampling activities, the sample logbook and COC forms will be transmitted to the CTO Manager for storage in project files. The original COC/analytical request form shall be submitted by the laboratory along with the data delivered. Any changes to the analytical requests that are required shall be made in writing to the laboratory. A copy of this written change shall be sent to the data validators and placed in the project files. The reason for the change shall be included in the project files so that recurring problems can be easily identified.

## 7.0 References and Attachments

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: [http://www.epa.gov/-fedfac/pdf/ufp\\_qapp\\_v1\\_0305.pdf](http://www.epa.gov/-fedfac/pdf/ufp_qapp_v1_0305.pdf).

Attachment 1: Chain-of-Custody Seal

Attachment 2: Generic Chain-of-Custody/Analytical Request Form

Author	Reviewer	Revisions (Technical or Editorial)
Tina Cantwell QA Officer	Ben Brantley Project Manager	Rev 0 — Initial Issue

**Attachment 1**  
**Chain-of-Custody Seal**

EXAMPLE CHAIN-OF-CUSTODY SEAL

[LABORATORY]	SAMPLE NO.	DATE	SEAL BROKEN BY
	SIGNATURE		DATE
	PRINT NAME AND TITLE ( <i>Inspector, Analyst or Technician</i> )		

**Attachment 2**  
**Example Chain-of-Custody/Analytical Request Form**

[illegible]

(1) AA=Ambient air, AQ=Air quality control, ASB=Asbestos, CK=Caulk, DS=Storm drain sediment, GS=Soil gas, IC=IDW Concrete, IDD=IDW Solid, IDS=IDW soil, IDW=IDW Water, LF=Free Product, MA=Mastic, PC=Paint Chips, SC=Cement/Concrete, SD=Sediment, SL=Sludge, SO=Soil, SQ=Soil/Solid quality control, SSD=Subsurface sediment, SU=Surface soil (<6 in), SW=Swab or wipe, TA=Animal tissue, TP=Plant tissue, TQ=Tissue quality control, WG=Ground water, WL=Leachate, WO=Ocean water, WP=Drinking water, WQ=Water quality control, WR=Ground water effluent, WS=Surface water, WU=Storm water, WW=Waste water

(2) Sample Type: AB=Ambient Bk, EB=Equipment Bk, FB=Field Bk, FD=Field Duplicate Sample, IDW=Investigative-Derived Waste, MIS=Incremental Sampling Methodology, N=Normal Environmental Sample, TB=Trip Bk

(3) Preservative added: HA=Hydrochloric Acid, NI=Nitric Acid, SH=Sodium Hydroxide, SA=Sulfuric Acid, ME=Methanol, SB=sodium bisulfate, ST=Sodium Thiosulfate. IF NO preservative added leave blank

Rev 5/12



# Equipment Decontamination

## Procedure 3-06

### 1.0 Purpose and Scope

- 1.1 This standard operating procedure (SOP) describes methods of equipment decontamination, to be used for activities where samples for chemical analysis are collected or where equipment will need to be cleaned before leaving the site or before use in subsequent activities.
- 1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

### 2.0 Safety

It is the responsibility of the **Site Safety Officer (SSO)** to set up the site zones (i.e., exclusion, transition, and clean) and decontamination areas. Generally the decontamination area is located within the transition zone, upwind of intrusive activities, and serves as the washing area for both personnel and equipment to minimize the spread of contamination into the clean zone. Typically, for equipment, a series of buckets are set up on a visqueen-lined bermed area. Separate spray bottles containing cleaning solvents as described in this procedure or the Contract Task Order (CTO) Work Plan (WP) and distilled water are used for final rinsing of equipment. Depending on the nature of the hazards and the site location, decontamination of heavy equipment, such as augers, pump drop pipe, and vehicles, may be accomplished using a variety of techniques.

All **Field Personnel** responsible for equipment decontamination must adhere to the site-specific health and safety plan (HSP) and must wear the personal protective equipment (PPE) specified in the site-specific HSP. Generally this includes, at a minimum, Tyvek® coveralls, steel-toed boots with boot covers or steel-toed rubber boots, safety glasses, American National Standards Institute-standard hard hats, and hearing protection (if heavy equipment is in operation). Air monitoring by the **SSO** may result in an upgrade to the use of respirators and cartridges in the decontamination area; therefore, this equipment must be available on site. If safe alternatives are not achievable, discontinue site activities immediately.

In addition to the aforementioned precautions, the following sections describe safe work practices that will be employed.

#### 2.1 Chemical Hazards associated with Equipment Decontamination

- Avoid skin contact with and/or incidental ingestion of decontamination solutions and water.
- Utilize PPE as specified in the site-specific HSP to maximize splash protection.
- Refer to material safety data sheets, safety personnel, and/or consult sampling personnel regarding appropriate safety measures (i.e., handling, PPE including skin and respiratory).
- Take the necessary precautions when handling detergents and reagents.

#### 2.2 Physical Hazards associated with Equipment Decontamination

- To avoid possible back strain, it is recommended to raise the decontamination area 1 to 2 feet above ground level.
- To avoid heat stress, over exertion, and exhaustion, it is recommended to rotate equipment decontamination among all site personnel.

- Take necessary precautions when handling field sampling equipment.

### **3.0 Terms and Definitions**

None.

### **4.0 Training and Qualifications**

- 4.1 The **CTO Manager** is responsible for ensuring that decontamination activities comply with this procedure. The **CTO Manager** is responsible for ensuring that all personnel involved in equipment decontamination shall have the appropriate education, experience, and training to perform their assigned tasks.
- 4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.
- 4.3 The **Field Manager** is responsible for ensuring that all field equipment is decontaminated according to this procedure.
- 4.4 All **Field Personnel** are responsible for the implementation of this procedure.

### **5.0 Procedure**

Decontamination of equipment used in soil/sediment sampling, groundwater monitoring, well drilling and well development, as well as equipment used to sample groundwater, surface water, sediment, waste, wipe, asbestos, and unsaturated zone, is necessary to prevent cross-contamination and to maintain the highest integrity possible in collected samples. Planning a decontamination program requires consideration of the following factors:

- Location where the decontamination procedures will be conducted
- Types of equipment requiring decontamination
- Frequency of equipment decontamination
- Cleaning technique and types of cleaning solutions appropriate to the contaminants of concern
- Method for containing the residual contaminants and wash water from the decontamination process
- Use of a quality control measure to determine the effectiveness of the decontamination procedure

The following subsections describe standards for decontamination, including the frequency of decontamination, cleaning solutions and techniques, containment of residual contaminants and cleaning solutions, and effectiveness.

#### **5.1 Decontamination Area**

Select an appropriate location for the decontamination area at a site based on the ability to control access to the area, the ability to control residual material removed from equipment, the need to store clean equipment, and the ability to restrict access to the area being investigated. Locate the decontamination area an adequate distance away and upwind from potential contaminant sources to avoid contamination of clean equipment.

#### **5.2 Types of Equipment**

Drilling equipment that must be decontaminated includes drill bits, auger sections, drill-string tools, drill rods, split barrel samplers, tremie pipes, clamps, hand tools, and steel cable. Decontamination of monitoring well development and groundwater sampling equipment includes submersible pumps, bailers, interface probes, water level meters, bladder pumps, airlift pumps, peristaltic pumps, and lysimeters. Other sampling equipment that requires decontamination includes, but is not limited to, hand trowels,

hand augers, slide hammer samplers, shovels, stainless-steel spoons and bowls, soil sample liners and caps, wipe sampling templates, composite liquid waste samplers, and dippers. Equipment with a porous surface, such as rope, cloth hoses, and wooden blocks, cannot be thoroughly decontaminated and shall be properly disposed of after one use.

### 5.3 **Frequency of Equipment Decontamination**

Decontaminate down-hole drilling equipment and equipment used in monitoring well development and purging prior to initial use and between each borehole or well. Down-hole drilling equipment, however, may require more frequent cleaning to prevent cross-contamination between vertical zones within a single borehole. When drilling through a shallow contaminated zone and installing a surface casing to seal off the contaminated zone, decontaminate the drilling tools prior to drilling deeper. Initiate groundwater sampling by sampling groundwater from the monitoring well where the least contamination is suspected. Decontaminate groundwater, surface water, and soil sampling devices prior to initial use and between collection of each sample to prevent the possible introduction of contaminants into successive samples.

### 5.4 **Cleaning Solutions and Techniques**

Decontamination can be accomplished using a variety of techniques and fluids. The preferred method of decontaminating major equipment, such as drill bits, augers, drill string, and pump drop-pipe, is steam cleaning. To steam clean, use a portable, high-pressure steam cleaner equipped with a pressure hose and fittings. For this method, thoroughly steam wash equipment and rinse it with potable tap water to remove particulates and contaminants.

A rinse decontamination procedure is acceptable for equipment such as bailers, water level meters, new and re-used soil sample liners, and hand tools. The decontamination procedure shall consist of the following: (1) wash with a non-phosphate detergent (Alconox®, Liquinox®, or other suitable detergent) and potable water solution; (2) rinse with potable water; (3) spray with laboratory-grade isopropyl alcohol; (4) rinse with deionized or distilled water; and (5) spray with deionized or distilled water. If possible, disassemble equipment prior to cleaning. Add a second wash at the beginning of the process if equipment is very soiled.

Decontaminating submersible pumps requires additional effort because internal surfaces become contaminated during usage. Decontaminate these pumps by washing and rinsing the outside surfaces using the procedure described for small equipment or by steam cleaning. Decontaminate the internal surfaces by recirculating fluids through the pump while it is operating. This recirculation may be done using a relatively long (typically 4 feet) large-diameter pipe (4-inch or greater) equipped with a bottom cap. Fill the pipe with the decontamination fluids, place the pump within the capped pipe, and operate the pump while recirculating the fluids back into the pipe. The decontamination sequence shall include: (1) detergent and potable water; (2) potable water rinse; (3) potable water rinse; and (4) deionized water rinse. Change the decontamination fluids after each decontamination cycle.

Solvents other than isopropyl alcohol may be used, depending upon the contaminants involved. For example, if polychlorinated biphenyls or chlorinated pesticides are contaminants of concern, hexane may be used as the decontamination solvent; however, if samples are also to be analyzed for volatile organics, hexane shall not be used. In addition, some decontamination solvents have health effects that must be considered. Decontamination water shall consist of distilled or deionized water. Steam-distilled water shall not be used in the decontamination process as this type of water usually contains elevated concentrations of metals. Decontamination solvents to be used during field activities will be specified in the CTO WP.

Rinse equipment used for measuring field parameters, such as pH (indicates the hydrogen ion concentration – acidity or basicity), temperature, specific conductivity, and turbidity with deionized or distilled water after each measurement. Also wash new, unused soil sample liners and caps with a fresh

detergent solution and rinse them with potable water followed by distilled or deionized water to remove any dirt or cutting oils that might be on them prior to use.

## 5.5 **Containment of Residual Contaminants and Cleaning Solutions**

A decontamination program for equipment exposed to potentially hazardous materials requires a provision for catchment and disposal of the contaminated material, cleaning solution, and wash water.

When contaminated material and cleaning fluids must be contained from heavy equipment, such as drill rigs and support vehicles, the area must be properly floored, preferably with a concrete pad that slopes toward a sump pit. If a concrete pad is impractical, planking can be used to construct solid flooring that is then covered by a nonporous surface and sloped toward a collection sump. If the decontamination area lacks a collection sump, use plastic sheeting and blocks or other objects to create a bermed area for collection of equipment decontamination water. Situate items, such as auger flights, which can be placed on metal stands or other similar equipment, on this equipment during decontamination to prevent contact with fluids generated by previous equipment decontamination. Store clean equipment in a separate location to prevent recontamination. Collect decontamination fluids contained within the bermed area and store them in secured containers as described below.

Use wash buckets or tubs to catch fluids from the decontamination of lighter-weight drilling equipment and hand-held sampling devices. Collect the decontamination fluids and store them on site in secured containers, such as U.S. Department of Transportation-approved drums, until their disposition is determined by laboratory analytical results. Label containers in accordance with Procedure 3-05, *IDW Management*.

## 6.0 **Quality Control and Assurance**

A decontamination program must incorporate quality control measures to determine the effectiveness of cleaning methods. Quality control measures typically include collection of equipment blank samples or wipe testing. Equipment blanks consist of analyte-free water that has been poured over or through the sample collection equipment after its final decontamination rinse. Wipe testing is performed by wiping a cloth over the surface of the equipment after cleaning. These quality control measures provide "after-the-fact" information that may be useful in determining whether or not cleaning methods were effective in removing the contaminants of concern.

## 7.0 **Records, Data Analysis, Calculations**

Any project where sampling and analysis is performed shall be executed in accordance with an approved sampling and analysis plan. This procedure may be incorporated by reference or may be incorporated with modifications described in the plan.

Deviations from this procedure or the sampling and analysis plan shall be documented in field records. Significant changes shall be approved by the **Program Quality Manager**.

## 8.0 **Attachments or References**

- 8.1 ASTM Standard D5088. 2008. *Standard Practice for Decontamination of Field Equipment Used at Waste Sites*. ASTM International, West Conshohocken, PA. 2008. DOI: 10.1520/D5088-02R08. [www.astm.org](http://www.astm.org).
- 8.2 NAVSEA T0300-AZ-PRO-010. *Navy Environmental Compliance Sampling and Field Testing Procedures Manual*. August 2009.
- 8.3 Procedure 3-05, *IDW Management*.

Author	Reviewer	Revisions (Technical or Editorial)
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue



**Standard Operating Procedure SOP-3-08 (MS)**  
**Soil Classification**

## **1.0 PURPOSE**

This section sets forth standard operating procedures for soil classification. If there are procedures from Resolution Consultants, state and/or federal that are not addressed in this Standard Operating Procedure (SOP) and are applicable to soil and rock classification then those procedures may be added as an appendix to the project specific Sampling and Analysis Plan (SAP).

## **2.0 SCOPE**

This procedure shall serve as management-approved professional guidance and is consistent with protocol in the Uniform Federal Policy-Quality Assurance Project Plan (DoD 2005). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved by either the Contract Task Order (CTO) Manager or the Quality Assurance (QA) Manager, and documented.

## **3.0 DEFINITIONS**

None.

## **4.0 RESPONSIBILITIES**

The CTO Manager, or designee, is responsible for ensuring that these standard soil and rock classification procedures are followed and that a qualified individual conducts or supervises the projects. A qualified individual is defined as a person with a degree in geology, hydrogeology, soil science, or geotechnical/civil engineering with at least 1 year of experience classifying soil. Supervision is defined as onsite and continuous monitoring of the individual conducting soil classification. The CTO Manager is responsible for ensuring that all personnel involved in soil and rock classification shall have the appropriate education, experience, and training to perform their assigned tasks. The CTO Manager is responsible for reviewing copies of the field boring log forms on a monthly basis at a minimum.

QA Manager or Technical Director is responsible for ensuring overall compliance with this procedure.

The Field Manager is responsible for ensuring that all project field staff follow these procedures.

Field personnel are responsible for the implementation of this procedure.

## **5.0 SOIL CLASSIFICATION**

The basic purpose of the classification of soil is to thoroughly describe the physical characteristics of the sample and to classify it according to an appropriate soil classification system. The Unified Soil Classification System (USCS) was developed so that soils could be described on a common basis by different investigators and serve as a "shorthand" description of soil. A classification of a soil in accordance with the USCS includes not only a group symbol and name, but also a complete word description.

Describing soil on a common basis is essential so that soil described by different site qualified personnel is comparable. Site individuals describing soil as part of site activities *must* use the classification system described herein to provide the most useful geologic database for all present and future subsurface investigations and remedial activities.

The site geologist or other qualified individual shall describe the soil and record the description in a boring log or logbook. The essential items in any written soil description are as follows:

- Classification group name (e.g., silty sand)
- Color, moisture, and odor
- Range of particle sizes and maximum particle size
- Approximate percentage of boulders, cobbles, gravel, sand, and fines
- Plasticity characteristics of the fines
- In-place conditions, such as consistency, density, and structure
- USCS classification symbol

The USCS serves as "shorthand" for classifying soil into 15 basic groups:

- GW<sup>1</sup> Well graded (poorly sorted) gravel (>50 percent gravel, <5percent fines)
- GP<sup>1</sup> Poorly graded (well sorted) gravel (>50percent gravel, <5percent fines)
- GM<sup>1</sup> Silty gravel (>50 percent gravel, >15 percent silt)
- GC<sup>1</sup> Clayey gravel (>50 percent gravel, >15 percent clay)
- SW<sup>1</sup> Well graded (poorly sorted) sand (>50 percent sand, <5 percent fines)
- SP<sup>1</sup> Poorly graded (well sorted) sand (>50 percent sand, <5 percent fines)

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<sup>1</sup> If percentage of fine is 5 percent to 15 percent, a dual identification shall be given (e.g., a soil with more than 50 percent poorly sorted gravel and 10 percent clay is designated GW-GC.

- SM<sup>1</sup> Silty sand (>50 percent sand, >15 percent silt)
- SC<sup>1</sup> Clayey sand (>50 percent sand, >15 percent clay)
- ML<sup>2</sup> Inorganic, low plasticity silt (slow to rapid dilatancy, low toughness, and plasticity)
- CL<sup>2</sup> Inorganic, low plasticity (lean) clay (no or slow dilatancy, medium toughness and plasticity)
- MH<sup>2</sup> Inorganic elastic silt (no to slow dilatancy, low to medium toughness and plasticity)
- CH<sup>2</sup> Inorganic, high plasticity (fat) clay (no dilatancy, high toughness, and plasticity)
- OL Organic low plasticity silt or organic silty clay
- OH Organic high plasticity clay or silt
- PT Peat and other highly organic soil

Figure 1 defines the terminology of the USCS. Flow charts presented in Figure 2 and Figure 3 indicate the process for describing soil. The particle size distribution and the plasticity of the fines are the two properties of soil used for classification. In some cases, it may be appropriate to use a borderline classification (e.g., SC/CL) if the soil has been identified as having properties that do not distinctly place the soil into one group.
















### **5.1 Estimation of Particle Size Distribution**

One of the most important factors in classifying a soil is the estimated percentage of soil constituents in each particle size range. Being proficient in estimating this factor requires extensive practice and frequent checking. The steps involved in determining particle size distribution are listed below:

1. Select a representative sample (approximately 1/2 of a 6-inch long by 2.5-inch diameter sample liner).
2. Remove all particles larger than 3 inches from the sample. Estimate and record the percent by volume of these particles. Only the fraction of the sample smaller than 3 inches is classified.

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<sup>2</sup> If the soil is estimated to have 15 percent to 25 percent sand or gravel, or both, the words "with sand" or "with gravel" (whichever predominates) shall be added to the group name (e.g., clay with sand, CL; or silt with gravel, ML). If the soil is estimated to have 30 percent or more sand or gravel, or both, the words "sandy" or "gravely" (whichever predominates) shall be added to the group name (e.g., sandy clay, CL). If the percentage of sand is equal to the percent gravel, use "sandy."

DEFINITION OF TERMS						
MAJOR DIVISIONS		SYMBOLS		TYPICAL DESCRIPTIONS		
COARSE GRAINED SOILS More Than Half of Material is Larger Than No. 200 Sieve Size	GRAVELS More Than Half of Coarse Fraction is Smaller Than No. 4 Sieve	CLEAN GRAVELS (Less than 6% Fines)		GW	Well graded gravels, gravel-sand mixtures, little or no fines	
				GP	Poorly graded gravels, gravel-sand mixtures, little or no fines	
		GRAVELS With Fines		GM	Silty gravels, gravel-sand-silt mixtures, non-plastic fines	
				GC	Clayey gravels, gravel-sand-clay mixtures, plastic fines	
	SANDS More Than Half of Coarse Fraction is Smaller Than No. 4 Sieve	CLEAN SANDS (Less than 6% Fines)		SW	Well graded sands, gravelly sands, little or no fines	
				SP	Poorly graded sands, gravelly sands, little or no fines	
		SANDS With Fines		SM	Silty sands, sand-silt mixtures, non-plastic fines	
				SC	Clayey sands, sand-clay mixtures, plastic fines	
FINE GRAINED SOILS More Than Half of Material is Smaller Than No. 200 Sieve Size	SILTS AND CLAYS Liquid Limit is Less Than 50%		ML	Inorganic silts, rock flour, fine sandy silts or clays, and clayey silts with non- or slightly-plastic fines		
			CL	Inorganic clays of low to medium plasticity, gravelly clays, silty clays, sandy clays, lean clays		
			OL	Organic silts and organic silty clays of low plasticity		
	SILTS AND CLAYS Liquid Limit is Greater Than 50%		MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts, clayey silt		
			CH	inorganic clays of high plasticity, fat clays		
			OH	Organic clays of medium to high plasticity, organic silts		
HIGHLY ORGANIC SOILS			PT	Peat and other highly organic soils		

GRAIN SIZES							
SILTS AND CLAYS	SAND			GRAVEL		COBBLES	BOULDERS
	FINE	MEDIUM	COARSE	FINE	COARSE		
	200	40	10	4	3/4"	3"	12"
U.S. STANDARD SERIES SIEVE				CLEAR SQUARE SIEVE OPENINGS			

**Figure 1: Unclassified Soil Classification System (USCS)**



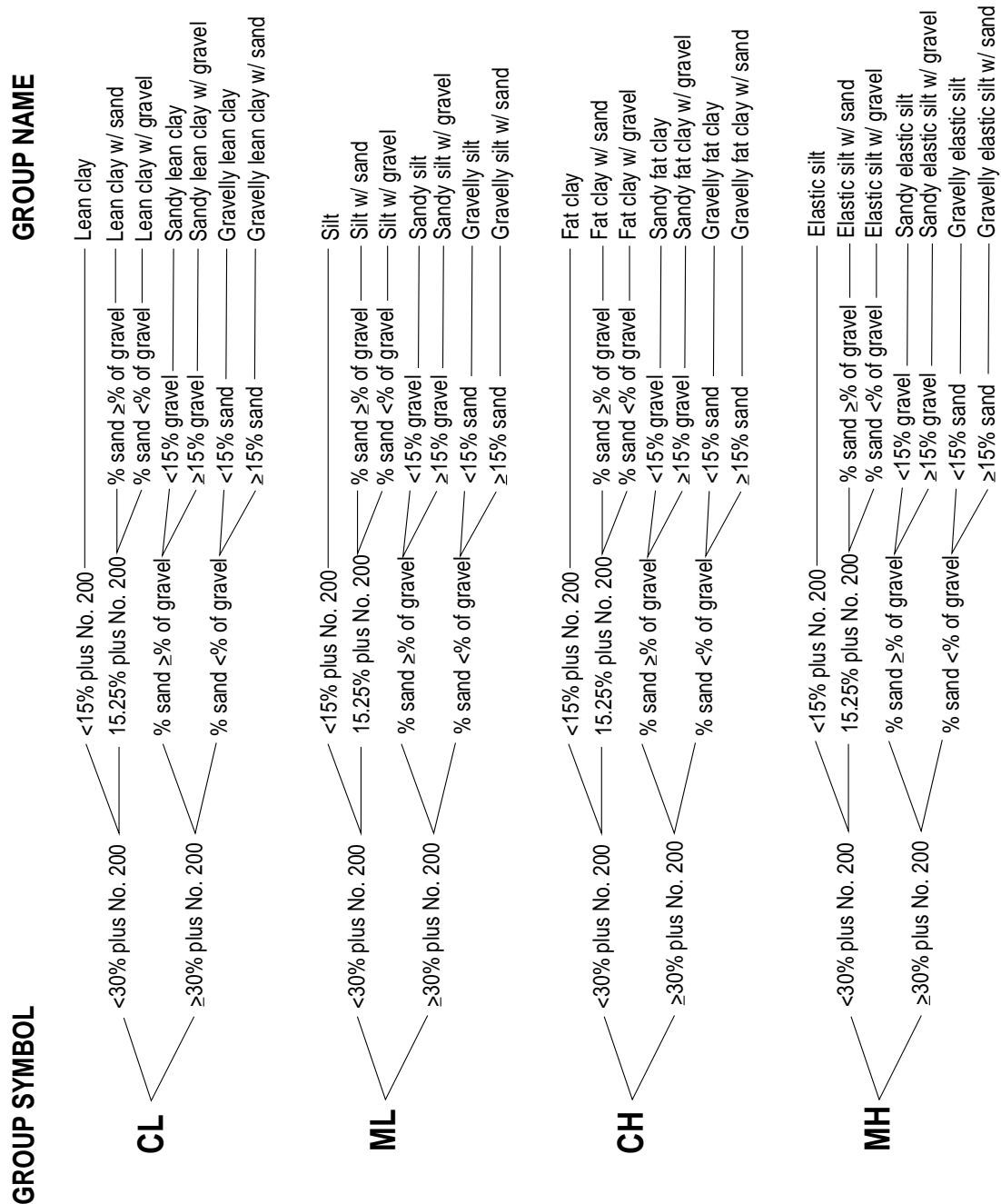
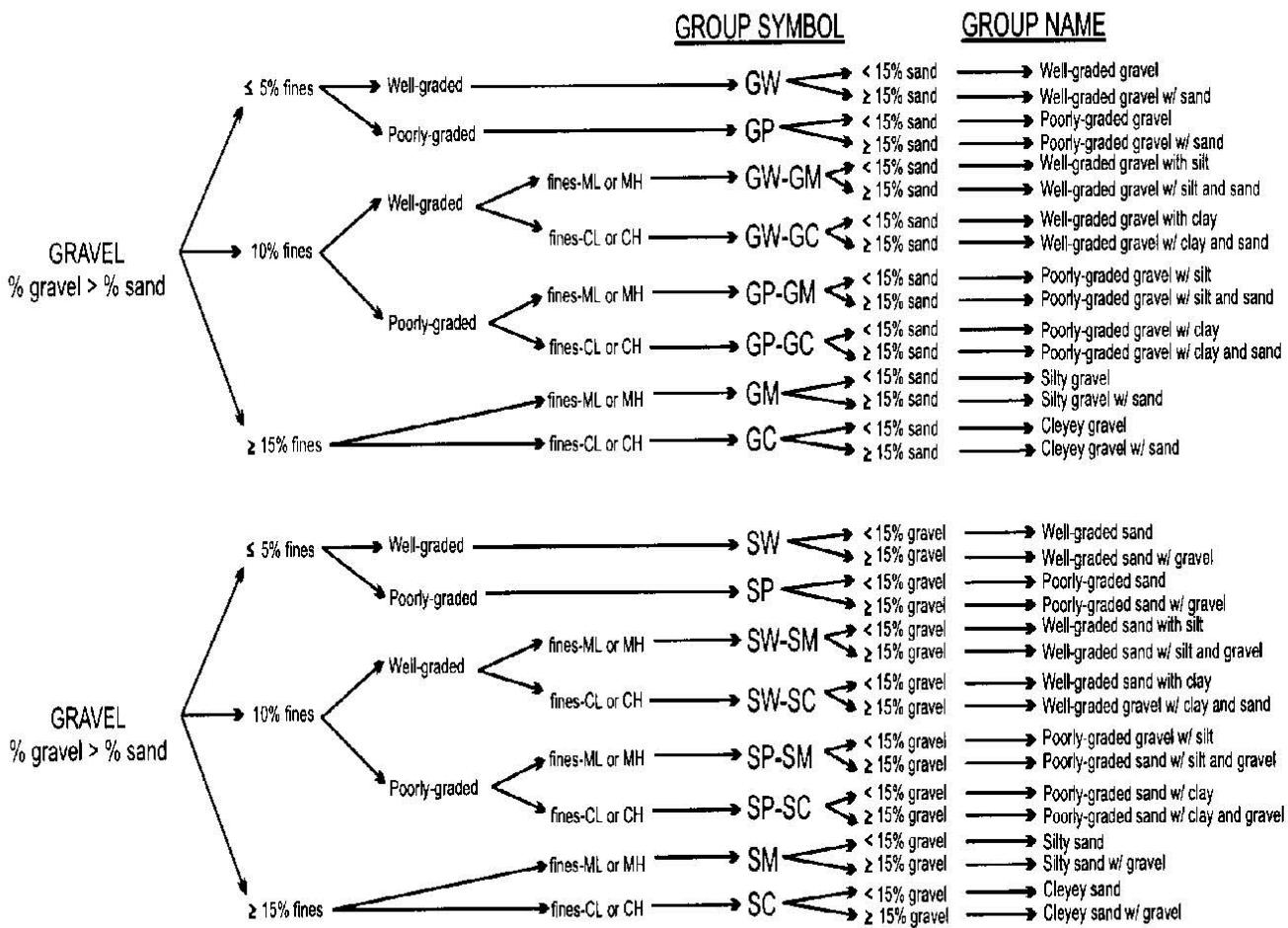


Figure 2: Flow Chart for Fine Grain Soil Classification





### Figure 3: Flow Chart for Soil with Gravel

3. Estimate and record the percentage of dry mass of gravel (less than 3 inches and greater than 1/4 inch).
4. Considering the rest of the sample, estimate, and record the percentage of dry mass of sand particles (about the smallest particle visible to the unaided eye).
5. Estimate and record the percentage of dry mass of fines in the sample (do not attempt to separate silts from clays).
6. Estimate percentages to the nearest 5 percent. If one of the components is present in a quantity considered less than 5 percent, indicate its presence by the term "trace".
7. The percentages of gravel, sand, and fines must add up to 100 percent. "Trace" is not included in the 100 percent total.

## **5.2 Soil Dilatancy, Toughness, and Plasticity**

### **5.2.1 Dilatancy**

To evaluate dilatancy, follow these procedures:

1. From the specimen, select enough material to mold into a ball about 1/2 inch (12 millimeters [mm]) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.
2. Smooth the soil ball in the palm of one hand with the blade of a knife or small spatula. Shake horizontally, striking the side of the hand vigorously against the other hand several times. Note the reaction of water appearing on the surface of the soil. Squeeze the sample by closing the hand or pinching the soil between the fingers, and note the reaction as none, slow, or rapid in accordance with the criteria in Table 1. The reaction is the speed with which water appears while shaking, and disappears while squeezing.

**Table 1  
Criteria for Describing Dilatancy**

<b>Description</b>	<b>Criteria</b>
None	No visible change in specimen.
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing.
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing.

### 5.2.2 Toughness

Following the completion of the dilatancy test, shape the test specimen into an elongated pat and roll it by hand on a smooth surface or between the palms into a thread about 1/8 inch (3 mm) in diameter. (If the sample is too wet to roll easily, spread it into a thin layer and allow it to lose some water by evaporation.) Fold the sample threads and re-roll repeatedly until the thread crumbles at a diameter of about 1/8 inch. The thread will crumble at a diameter of 1/8 inch when the soil is near the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread. After the thread crumbles, lump the pieces together and knead it until the lump crumbles. Note the toughness of the material during kneading. Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in Table 2.

**Table 2  
Criteria for Describing Toughness**

<b>Description</b>	<b>Criteria</b>
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft.
Medium	Medium pressure is required to roll the thread near the plastic limit. The thread and the lump have medium stiffness.
High	Considerable pressure is required to roll the thread near the plastic limit. The thread and the lump have very high stiffness.

### 5.2.3 Plasticity

The plasticity of a soil is defined by the ability of the soil to deform without cracking, the range of moisture content over which the soil remains in a plastic state, and the degree of cohesiveness at the plastic limit. The plasticity characteristic of clays and other cohesive materials is defined by the liquid limit and plastic limit. The liquid limit is defined as the soil moisture content at which soil passes from the liquid to the plastic state as moisture is removed. The test for the liquid limit is a laboratory, not a field, analysis.

The plastic limit is the soil moisture content at which a soil passes from the plastic to the semi-solid state as moisture is removed. The plastic limit test can be performed in the field and is indicated by the ability to roll a 1/8-inch (0.125-inch) diameter thread of fines, the time required to roll the thread, and the number of times the thread can be re-rolled when approaching the plastic limit.

The plasticity tests are not based on natural soil moisture content, but on soil that has been thoroughly mixed with water. If a soil sample is too dry in the field, add water prior to performing

classification. If a soil sample is too sticky, spread the sample thin and allow it to lose some soil moisture.

Table 3 presents the criteria for describing plasticity in the field using the rolled thread method.

**Table 3  
Criteria for Describing Plasticity**

Description	Criteria
Non-Plastic	A 1/8-inch thread cannot be rolled.
Low Plasticity	The thread can barely be rolled.
Medium Plasticity	The thread is easy to roll and not much time is required to reach the plastic limit.
High Plasticity	It takes considerable time rolling the thread to reach the plastic limit.

#### 5.2.4 Angularity

The following criteria describe the angularity of the coarse sand and gravel particles:

- **Rounded** particles have smoothly-curved sides and no edges.
- **Subrounded** particles have nearly plane sides, but have well-rounded corners and edges.
- **Subangular** particles are similar to angular, but have somewhat rounded or smooth edges.
- **Angular** particles have sharp edges and relatively plane sides with unpolished surfaces. Freshly broken or crushed rock would be described as angular.

#### 5.2.5 Color, Moisture, and Odor

The natural moisture content of soil is very important. Table 4 shows the terms for describing the moisture condition and the criteria for each.

**Table 4  
Soil Moisture Content Qualifiers**

Qualifier	Criteria
Dry	Absence of moisture, dry to the touch
Moist	Damp but no visible water
Wet	Visible water, usually soil is below water table



Color is described by hue and chroma using the Munsell Soil Color Chart (Munsell 2000). For uniformity, all site geologists shall utilize this chart for soil classification. Doing so will facilitate correlation of geologic units between boreholes logged by different geologists. The Munsell Color Chart is a small booklet of numbered color chips with names like "5YR 5/6, yellowish-red." Note mottling or banding of colors. It is particularly important to note and describe staining because it may indicate contamination.

In general, wear a respirator if strong organic odors are present. If odors are noted, describe them if they are unusual or suspected to result from contamination. An organic odor may have the distinctive smell of decaying vegetation. Unusual odors may be related to hydrocarbons, solvents, or other chemicals in the subsurface. An organic vapor analyzer may be used to detect the presence of volatile organic contaminants.

#### **5.2.6 In-Place Conditions**

Describe the conditions of undisturbed soil samples in terms of their density/consistency (i.e., compactness), cementation, and structure utilizing the following guidelines:

##### **5.2.6.1 Density/Consistency**

Density and consistency describe a physical property that reflects the relative resistance of a soil to penetration. The term "density" is commonly applied to coarse to medium-grained sediments (i.e., gravels, sands), whereas the term "consistency" is normally applied to fine-grained sediments (i.e., silts, clays). There are separate standards of measure for both density and consistency that are used to describe the properties of a soil.

The density or consistency of a soil is determined by observing the number of blows required to drive a 1 3/8-inch (35 mm) diameter split barrel sampler 18 inches using a drive hammer weighing 140 lbs (63.5 kilograms [kg]) dropped over a distance of 30 inches (0.76 meters). Record the number of blows required to penetrate each 6 inches of soil in the field boring log during sampling. The first 6 inches of penetration is considered to be a seating drive; therefore, the blow count associated with this seating drive is recorded, but not used in determining the soil density/consistency. The sum of the number of blows required for the second and third 6 inches of penetration is termed the "standard penetration resistance," or the "N-value." The observed number of blow counts must be corrected by an appropriate factor if a different type of sampling device (e.g., Modified California Sampler with liners) is used. For a 2 3/8-inch inner diameter (I.D.) Modified California Sampler equipped with brass or stainless steel liners and penetrating a

cohesionless soil (sand/gravel), the N-value from the Modified California Sampler must be divided by 1.43 to provide data that can be compared to the 1 3/8-inch diameter sampler data.

For a cohesive soil (silt/clay), the N-value for the Modified California Sampler should be divided by a factor of 1.13 for comparison with 1 3/8-inch diameter sampler data.

Drive the sampler and record blow counts for each 6-inch increment of penetration until one of the following occurs:

- A total of 50 blows have been applied during any one of the three 6-inch increments; a 50-blow count occurrence shall be termed "refusal" and noted as such on the boring log.
- A total of 150 blows have been applied.
- The sampler is advanced the complete 18 inches without the limiting blow counts occurring, as described above.

If the sampler is driven less than 18 inches, record the number of blows per partial increment on the boring log. If refusal occurs during the first 6 inches of penetration, the number of blows will represent the N-value for this sampling interval. Table 5 and Table 6 present representative descriptions of soil density/consistency vs. N-values.

**Table 5**  
**Measuring Soil Density with a California Sampler — Relative Density (Sands, Gravels)**  
**Field Criteria (N-Value)**

<b>Description</b>	<b>1 3/8 in. ID Sampler</b>	<b>2 in. ID Sampler using 1.43 factor</b>
Very Loose	0–4	0–6
Loose	4–10	6–14
Medium Dense	10–30	14–43
Dense	30–50	43–71
Very Dense	> 50	> 71

**Table 6**  
**Measuring Soil Density with a California Sampler — Fine Grained Cohesive Soil**

Description	Field Criteria (N-Value)	
	1 3/8 in. ID Sampler	2 in. ID Sampler using 1.13 factor
Very Soft	0–2	0–2
Soft	2–4	2–4
Medium Stiff	4–8	4–9
Stiff	8–16	9–18
Very Stiff	16–32	18–36
Hard	> 32	> 36

For undisturbed fine-grained soil samples, it is also possible to measure consistency with a hand-held penetrometer. The measurement is made by placing the tip of the penetrometer against the surface of the soil contained within the sampling liner or Shelby tube, pushing the penetrometer into the soil a distance specified by the penetrometer manufacturer, and recording the pressure resistance reading in pounds per square foot (psf). The values are as follows (Table 7):

**Table 7**  
**Measuring Soil Consistency with a Hand-Held Penetrometer**

Description	Pocket Penetrometer Reading (psf)
Very Soft	0–250
Soft	250–500
Medium Stiff	500–1000
Stiff	1000–2000
Very Stiff	2000–4000
Hard	>4000

Consistency can also be estimated using thumb pressure using Table 8.

**Table 8**  
**Measuring Soil Consistency Using Thumb Pressure**

Description	Criteria
Very Soft	Thumb will penetrate soil more than 1 inch (25 mm)
Soft	Thumb will penetrate soil about 1 inch (25 mm)
Firm	Thumb will penetrate soil about 1/4 inch (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very Hard	Thumbnail will not indent soil

#### **5.2.6.2 Cementation**

Cementation is used to describe the friability of a soil. Cements are chemical precipitates that provide important information as to conditions that prevailed at the time of deposition, or conversely, diagenetic effects that occurred following deposition. Seven types of chemical cements are recognized by Folk (1980). They are as follows:

1. Quartz — siliceous
2. Chert — chert-cemented or chalcedonic
3. Opal — opaline
4. Carbonate — calcitic, dolomitic, sideritic (if in doubt, calcareous should be used)
5. Iron oxides — hematitic, limonitic (if in doubt, ferruginous should be used)
6. Clay minerals — if the clay minerals are detrital or have formed by recrystallization of a previous clay matrix, they are not considered to be a cement. Only if they are chemical precipitates, filling previous pore space (usually in the form of accordion-like stacks or fringing radial crusts) should they be included as "kaolin-cemented," "chlorite-cemented," etc.
7. Miscellaneous minerals — pyritic, collophane-cemented, glauconite-cemented, gypsiferous, anhydrite-cemented, baritic, feldspar-cemented, etc.

The degree of cementation of a soil is determined qualitatively by utilizing finger pressure on the soil in one of the sample liners to disrupt the gross soil fabric. The three cementation descriptors are as follows:

1. Weak — friable; crumbles or breaks with handling or slight finger pressure
2. Moderate — friable; crumbles or breaks with considerable finger pressure
3. Strong — not friable; will not crumble or break with finger pressure

#### **5.2.6.3 Structure**

This variable is used to qualitatively describe physical characteristics of soil that are important to incorporate into hydrogeological and/or geotechnical descriptions of soil at a site. Appropriate soil structure descriptors are as follows:

- Granular — spherically shaped aggregates with faces that do not accommodate adjoining faces
- Stratified — alternating layers of varying material or color with layers at least 6 mm (1/4 inch) thick; note thickness
- Laminated — alternating layers of varying material or color with layers less than 6 mm (1/4 inch) thick; note thickness
- Blocky — cohesive soil that can be broken down into small angular or subangular lumps that resist further breakdown
- Lensed — inclusion of a small pocket of different soil, such as small lenses of sand, should be described as homogeneous if it is not stratified, laminated, fissured, or blocky. If lenses of different soil are present, the soil being described can be termed homogeneous if the description of the lenses is included
- Prismatic or Columnar — particles arranged about a vertical line, ped is bounded by planar, vertical faces that accommodate adjoining faces; prismatic has a flat top; columnar has a rounded top
- Platy — particles are arranged about a horizontal plane

#### **5.2.6.4 Other Features**

- Mottled — soil that appears to consist of material of two or more colors in blotchy distribution
- Fissured — breaks along definite planes of fracture with little resistance to fracturing (determined by applying moderate pressure to sample using thumb and index finger)

- Slickensided — fracture planes appear polished or glossy, sometimes striated (parallel grooves or scratches)

### **5.2.7 Development of Soil Description**

Develop standard soil descriptions according to the following examples. There are three principal categories under which all soil can be classified. They are described below.

#### **5.2.7.1 Coarse-grained Soil**

Coarse-grained soil is divided into sands and gravels. A soil is classified as a sand if over 50 percent of the coarse fraction is "sand-sized." It is classified as a gravel if over 50 percent of the coarse fraction is composed of "gravel-sized" particles.

The written description of a coarse-grained soil shall contain, in order of appearance: Typical name including the second highest percentage constituent as an adjective, if applicable (underlined); grain size of coarse fraction; Munsell color and color number; moisture content; relative density; sorting; angularity; other features, such as stratification (sedimentary structures) and cementation, possible formational name, primary USCS classification, secondary USCS classification (when necessary), and approximate percentages of minor constituents (i.e., sand, gravel, shell fragments, rip-up clasts) in parentheses.

Example: POORLY-SORTED SAND WITH SILT, medium- to coarse-grained, light olive gray, 5Y 6/2, saturated, loose, poorly sorted, subrounded clasts, SW/SM (minor silt with approximately 20 percent coarse-grained sand-sized shell fragments, and 80 percent medium-grained quartz sand, and 5 percent to 15 percent ML).

#### **5.2.7.2 Fine-grained Soil**

Fine-grained soil is further subdivided into clays and silts according to its plasticity. Clays are rather plastic, while silts have little or no plasticity.

The written description of a fine-grained soil should contain, in order of appearance: Typical name including the second highest percentage constituent as an adjective, if applicable (underlined); Munsell color; moisture content; consistency; plasticity; other features, such as stratification, possible formation name, primary USCS classification, secondary USCS classification (when necessary), and the percentage of minor constituents in parentheses.



Example: SANDY LEAN CLAY, dusky red, 2.5 YR 3/2, moist, firm, moderately plastic, thinly laminated, CL (70 percent fines, 30 percent sand, with minor amounts of disarticulated bivalves [about 5 percent]).

#### **5.2.7.3 Organic Soil**

For highly organic soil, describe the types of organic materials present as well as the type of soil constituents present using the methods described above. Identify the soil as an organic soil, OL/OH, if the soil contains enough organic particles to influence the soil properties. Organic soil usually has a dark brown to black color and may have an organic odor. Often, organic soils will change color, (e.g., from black to brown) when exposed to air. Some organic soils will lighten in color significantly when air-dried. Organic soils normally will not have a high toughness or plasticity. The thread for the toughness test will be spongy.

Example: ORGANIC CLAY, black, 2.5Y, 2.5/1, wet, soft, low plasticity, organic odor, OL (100 percent fines), weak reaction to HCl.

### **6.0 RECORDS**

Document soil classification information collected during soil sampling onto the field boring logs, field trench logs, and into the field notebook. Copies of this information shall be placed in the project files.

### **7.0 HEALTH AND SAFETY**

Observe standard health and safety practices according to the CTO-specific health and safety plan. Monitoring during excavation activities should determine contaminant concentrations and any required personal protective equipment (PPE) that may be necessary.

Suggested minimum protection during soil and rock classification activities in conjunction with field excavations shall include disposable nitrile gloves, steel-toed boots and overboots, safety glasses, hearing protection, and an American National Standards Institute-standard hard hat. Respirators and cartridges may be necessary depending on the contaminant concentrations and shall always be available on site. At no time during classification activities are personnel to reach for debris near machinery that is in operation, place any samples in their mouth, or come in contact with the soils/rocks without the use of gloves.

In addition to the aforementioned precautions, employ the following safe work practices:

**Physical Hazards Associated With Soil Classification:**

- To avoid lifting injuries associated with large specimens, use the large muscles of the legs, not the back.
- Be wary of uneven terrain to avoid slip/trip/fall conditions.
- To avoid heat/cold stress as a result of exposure to extreme temperatures and PPE, drink electrolyte replacement fluids (1-2 cups/hour is recommended) and, in cases of extreme cold, wear fitted insulating clothing.
- Be aware of restricted mobility due to PPE.

**8.0 REFERENCES**

American Society for Testing and Materials (ASTM). 2000. *Standard Practice for Description and Identification of Soils (Visual, Manual Procedure)*. D 2488-00. West Conshohocken, PA.

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: [http://www.epa.gov/fedfac/pdf/ufp\\_qapp\\_v1\\_0305.pdf](http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf).

Folk, Robert L. 1980. *Petrology of Sedimentary Rocks*. Austin, TX: Hemphill Publishing Company.

Munsell Color Company (Munsell). 2000. *Munsell Soil Color Chart*, (Revised). Baltimore.

**9.0 ATTACHMENTS**

None.

# Monitoring Well Installation

## Procedure 3-12

### 1.0 Purpose and Scope

- 1.1 This standard operating procedure (SOP) describes the methods to be used during the installation of groundwater monitoring wells. It describes the components of monitoring well design and installation and sets forth the rationale for use of various well installation techniques in specific situations.
- 1.2 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

### 2.0 Safety

- 2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the Contract Task Order (CTO) Work Plan (WP) and/or direction from the **Site Safety Officer (SSO)**.
- 2.2 Before well installation commences, appropriate entities (e.g. DigSafe, local public works departments, company facilities) must be contacted to assure the anticipated well locations are marked for utilities, including electrical, telecommunications, water, sewer, and gas.
- 2.3 Physical Hazards Associated with Well Installation
- Stay clear of all moving equipment and avoid wearing loose fitting clothing.
  - When using an approved retractable-blade knife, always cut away from one self and make sure there are no other people in the cutting path or the retractable-blade knife.
  - To avoid slip/trip/fall conditions during drilling activities, keep the area clear of excess soil cuttings and groundwater. Use textured boots/boot cover bottoms in muddy areas.
  - To avoid heat/cold stress as a result of exposure to extreme temperatures and personal protective equipment (PPE), drink electrolyte replacement fluids (1 to 2 cups per hour is recommended) and, in cases of extreme cold, wear fitted insulating clothing.
  - To avoid hazards associated with subsurface utilities, ensure all sampling locations have been properly surveyed as described in SOP 3-01, Utility Clearance.
  - Be aware of restricted mobility caused by PPE.

### 3.0 Terms and Definitions

- 3.1 **Annulus:** The annulus is the down-hole space between the borehole wall and the well casing and screen.
- 3.2 **Bridge:** A bridge is an obstruction in the drill hole or annulus. A bridge is usually formed by caving of the wall of the well bore, by the intrusion of a large boulder, or by the placement of filter pack materials during well completion. Bridging can also occur in the formation during well development.
- 3.3 **Filter Pack:** Filter pack is sand or gravel that is smooth, uniform, clean, well-rounded, and siliceous. It is placed in the annulus of the well between the borehole wall and the well screen to prevent formation materials from entering the well and to stabilize the adjacent formation.
- 3.4 **Grout:** Grout is a fluid mixture of cement and water that can be forced through a tremie pipe and emplaced in the annular space between the borehole and casing to form an impermeable seal. Various additives, such as sand, bentonite, and polymers, may be included in the mixture to meet certain requirements.
- 3.5 **Heaving (Running) Sands:** Loose sands in a confined water-bearing zone or aquifer which tend to rise up into the drill stem when the confining unit is breached by the drill bit. Heaving sands occur when the water in the aquifer has a pressure head great enough to cause upward flow into the drill stem with enough velocity to overcome the weight of the sand.
- 3.6 **Sieve Analysis:** Sieve analysis is the evaluation of the particle-size distribution of a soil, sediment, or rock by measuring the percentage of the particles that will pass through standard sieves of various sizes.

### 4.0 Interferences

- 4.1 Heaving sands may be problematic in unconsolidated sands encountered below the water table.
- 4.2 Rotary drilling methods requiring bentonite-based drilling fluids should be used with caution to drill boreholes that will be used for monitoring well installation. The bentonite mud builds up on the borehole walls as a filter cake and permeates the adjacent formation, potentially reducing the permeability of the material adjacent to the well screen.
- 4.3 If water or other drilling fluids have been introduced into the boring during drilling or well installation, samples of these fluids should be obtained and analyzed for chemical constituents that may be of interest at the site. In addition, an attempt should be made to recover the quantity of fluid or water that was introduced, either by flushing the borehole prior to well installation and/or by overpumping the well during development.
- 4.4 Track-mounted drill rigs are suitable for travelling on many types of landscapes that truck-mounted units cannot access, but may have limitations on extremely uneven or soft terrain.
- 4.5 Care should be taken to prevent cross-contamination between well locations. All drilling equipment coming in contact with potentially contaminated soil and/or groundwater will be decontaminated by the drilling subcontractor prior to initial drilling activities and between drilling locations in accordance with SOP 3-06, Equipment Decontamination.

### 5.0 Training and Qualifications

#### 5.1 Qualifications and Training

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

## 5.2 Responsibilities

- 5.2.1 **Contract Task Order (CTO) Managers** are responsible for issuing sampling and analysis plans (SAPs) that reflect the procedures and specifications presented in this procedure. Individual municipalities, county agencies, and possibly state regulatory agencies enforce regulations that may include well construction and installation requirements. The **CTO Manager** shall be familiar with current local and state regulations, and ensure that these regulations are followed. The **CTO Manager** is responsible for ensuring that all personnel involved in monitoring well installation shall have the appropriate education, experience, and training to perform their assigned tasks.
- 5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.
- 5.2.3 The **Field Manager** is responsible for direct supervision of the installation of monitoring wells and ensuring that procedures and specifications are implemented in the field in accordance with the approved SAP and well installation permits. The qualifications for the **Field Manager** must be in accordance with local jurisdictions with authority over the operations conducted.
- 5.2.4 All field personnel are responsible for the implementation of this procedure.
- 5.2.5 The on-site hydrogeologist/engineer is expected to obtain a description of the lithologic samples obtained during the excavation and construction of a monitoring well. These data are often required to provide guidance regarding the installation of specific components of the monitoring well. Guidance for lithologic sample collection and sample description is contained within SOP 3-16, Soil and Rock Classification.

## 6.0 Equipment and Supplies

- 6.1 Materials provided by the drilling contractor may include:
- Drill rig, drill rods, hollow stem augers, etc.
  - Decontamination equipment (e.g., steam cleaner, high-pressure washer, brushes, etc.)
  - Decontamination pad materials
  - Well screen/riser pipe with flush-threaded couplings including riser and bottom caps
  - Clean, filter sand
  - Bentonite chips or pellets
  - Cement grout and tremie pipe
  - Portland cement for well pad completion
  - Steel protective riser covers and locking caps
  - Weighted calibrated tape
  - Split-spoon samplers
  - 55-gallon drums or containers for drill cuttings, decontamination fluids, etc.
- 6.2 In addition to those materials provided by the drilling contractor, equipment and materials required by the project geologist/engineer may include, but is not limited to, the following:
- Photoionization Detector (PID)
  - Spill kit, including at a minimum sorbent pads and shovel (if not provided by subcontractor)

- Plastic sheeting
- Teaspoon or spatula
- Resealable plastic bags
- Boring Log Records
- Decontamination materials (per SOP No. 3-06 - Equipment Decontamination)
- Weighted measuring tape for depth measurement
- Soil logging materials (e.g. USCS classification field card, millimeter rule, hand lens, etc.)
- Survey lathes or pin flags
- Digital camera
- PPE as required by the HASP
- Planning documents including the site-specific HASP and SAP
- Large indelible ink or paint pen
- Field logbook/field forms/site maps (water proof)

## **7.0 Procedure**

### **7.1 General Procedures**

- Specific drilling, sampling, and installation equipment and methodology will be dictated by the type of well to be installed (e.g., single case (Type II), double case (Type III), bedrock, etc.), geologic characteristics of the site, the type of contaminants being monitored, and local and state regulations.
- For access to locations when travelling over difficult terrain, an appropriate line should be chosen before mobilizing the drill rig or other support vehicles. If clearing of trees or ground cover is required, perform these activities in advance to avoid down time. Avoid wet or soft areas where possible or use ground mats and/or timbers to aid in supporting the rig as it travels. If drilling on soft material, place geomatting and ground mats under the rig tracks or stabilizers prior to drilling.
- A utility locate must be conducted to identify all underground utilities at the site prior to drilling (refer to SOP 3-01, Utility Clearance). Proper clearance procedures for aboveground/overhead utilities must also be followed as specified in the HASP.
- Although new well materials (well screen and riser pipe) generally arrive at the site boxed and sealed within plastic bags, it is sometimes necessary to decontaminate the materials prior to their use. Well materials should be inspected by the project geologist/engineer upon delivery to check for cleanliness. If the well materials appear dirty, or if local or regional regulatory guidance requires decontamination, then well material decontamination should be performed by the drilling subcontractor in accordance with SOP 3-06, Equipment Decontamination.
- The diameter of the borehole must be a minimum of 2 inches greater than the outside diameter of the well screen or riser pipe used to construct the well. This is necessary so that sufficient annular space is available to install filter packs, bentonite seals, and grout seals, and allow the passage of tremie pipe where grouting at depth is required. Bedrock wells may require reaming after coring in order to provide a large enough borehole diameter for well installation.
- When soil sampling is required (refer to the SAP), soil samples will be collected for visual logging by advancing split-spoon samplers through the augers. The soil will be visually logged by a field geologist and include lithologic characteristics (i.e., soil type, color, density, moisture content, etc.) using the the



methods described in SOP 3-16, Soil and Rock Classification. This information will be recorded on a boring/well log form, along with well construction details.

## 7.2 Drilling Techniques

Drilling of monitoring well boreholes may be accomplished by a variety of methods as described below. Preferred methods include those that temporarily case the borehole during drilling (i.e., hollow stem auger and sonic methods) using an override system. Other methods can be used where specific subsurface conditions or well design criteria dictate.

- Hollow stem auger (HSA) – Borings are advanced by rotating steel hollow stem augers with an attached cutting head. Soil cuttings are displaced by the cutting head and transported to the surface via continuous spiral flights attached to each auger stem. This method is widely used for unconsolidated soils that have a tendency to collapse within the boring. A bottom plug can be placed in the bottom auger to prevent soils from entering and clogging the auger, especially in the case of heaving sands. However, a bottom plug cannot be used when soil samples are to be collected through the augers. Soil plugs that accumulate in the bottom of the auger must be removed or knocked out prior to sampling or well installation.
- Solid stem auger – This type of drilling method is similar to HSA drilling using a solid stem or sealed hollow stem auger flights to advance the boring. Solid stem, continuous flight auger use is limited to semi-consolidated sediments or to cohesive or semi-cohesive unconsolidated sediments that don't have a tendency to collapse when disturbed.
- Sonic methods – Sonic drilling consists of advancing concentric hollow drill casings (inner and outer) using rotation in conjunction with axial vibration of the drill casing. Once the casings are advanced to the appropriate depth, the inner string is removed with a core of drill cuttings while the outer casing remains in place to keep the borehole open. Cuttings are removed from the inner casing relatively intact for logging or sampling purposes. This drilling method is used for a variety of soil types, from heaving sands to consolidated or indurated formations. Smearing of the formation along the borehole walls is minimal since moderate vibration and rotation techniques are used to advance the casings. Since the total borehole diameter in sonic drilling is only incrementally larger than the inner casing diameter, care should be taken during installation of the monitoring well to ensure the well is centered and adequate space is available for annular materials.
- Rotary methods (water or mud) – Rotary drilling methods consist of drill rods coupled to a drill bit that rotates and cuts through the soils to advance the borehole. Water or drilling fluid ("mud") is forced through the hollow drill rods and drill bit as the rods are rotated. The soil cuttings are forced up the borehole with the drilling fluids to the surface and the fluids recirculated. The drilling fluid provides a hydrostatic pressure that reduces or prevents the borehole from collapsing. Clean, potable water must be used for water-rotary drilling to prevent introducing trace contaminants. A sample of the potable water should be collected during the course of well installation for analysis of the same parameters defined for the groundwater samples. If mud-rotary is used to advance boreholes, potable water and bentonite drilling mud should only be used. No chemical additives shall be mixed in the drilling fluid to alter viscosity or lubricating properties. Adequate well development is essential for removal of drilling mud and fluids from the formation materials and ensure collection of representative groundwater samples.
- Rotary methods (Air) – Air rotary methods are similar to water rotary but use high air velocities in place of drilling fluids to rotate the drill bit and carry the soil cuttings up the borehole to the surface. Care must be taken to ensure that contaminants are not introduced into the air stream from compressor oils, etc. Most compressor systems are compatible with a coalescing filter system. Cuttings exiting the borehole under pressure must be controlled, especially when drilling in a zone of potential contamination. This can be accomplished by using an air diverter with hose or pipe to carry the cuttings to a waste container. Letting the cuttings blow uncontrolled from the borehole is not acceptable.

### 7.3 Well Construction and Installation

- If rotary drilling techniques are used, the borehole should be flushed or blown free of material prior to well installation. If hollow stem augers are used, the soil or bottom plug should be removed and the augers raised approximately six inches above the bottom of the borehole, while slowly rotating the augers to remove cuttings from the bottom of the boring. The depth of the borehole should be confirmed with a weighted, calibrated tape.
- The riser pipe and screen should be connected with flush-threaded joints and assembled wearing clean, disposable gloves. No solvent or anti-seize compound should be used on the connections. The full length of the slotted portion of the well screen and unslotted riser pipe should be measured and these measurements recorded on a well construction form (Attachment 1).
- If placed in an open borehole, the assembled well should be carefully lowered and centered in the borehole so that the well is true, straight, and vertical throughout. Centering can also be accomplished with the use of centralizers, if necessary. However, centralizers should be placed so that they do not inhibit the installation of filter sand, bentonite seal, and annular grout. Wells less than 50 deep generally do not require centralizers.
- If hollow stem augers are used, the well should be lowered through the augers and each auger flight removed incrementally as the filter sand, bentonite seal, and grout are tremmied or poured into the annular space of the well. The well should be temporarily capped before filter sand and other annular materials are installed.
- Clean, silica sand should be placed around the well screen to at least 1 foot above the top of the screen. The filter sand should be appropriately graded and compatible with the selected screen size and surrounding formation materials. In general, the filter pack should not extend more than 3 feet above the top of the screen to limit the thickness of the monitoring zone. As the filter pack is placed, a weighted tape should be lowered in the annular space to verify the depth to the top of the layer. This measurement will be recorded on the well construction form (Attachment 1). If necessary, to eliminate possible bridging or creation of voids, placement of the sand pack may require the use of a tremie pipe. Tremie pipe sandpack installations are generally suggested for deeper wells and for wells which are screened some distance beneath the water table.
- A minimum 2-foot thick layer of bentonite pellets or slurry seal will be installed immediately above the filter sand to prevent vertical flow within the boring from affecting the screened interval. Bentonite chips/pellets must be hydrated if placed above the water table prior to grouting. If bridging is of concern as in the case of deep wells, powdered bentonite may be mixed with water into a very thick slurry and a tremie pipe used to place the seal to the desired depth. Placement of the bentonite seal in the borehole will be recorded on the well construction form (Attachment 1).
- The remaining annular space around the well will be grouted from the top of the bentonite seal to the surface with a grout composed of neat cement, a bentonite cement mixture, or high solids sodium bentonite grout.
- Each well riser will be secured with an expandable, locking cap (vented if possible). Optionally, a hole can be drilled in the upper portion of the riser to allow venting of the well.
- The well will be completed within a concrete well pad consisting of a Portland cement/sand mixture. Well pads are generally 3 feet by 3 feet square but may be larger or smaller depending on site conditions and state-specific well construction standards. Round concrete well pads are also acceptable. A minimum of 1 inch of the finished pad should be below grade to prevent washing and undermining by soil erosion.
- If completed as a flush-mount well, the well riser will be cut off approximately 4 to 6 inches below ground surface and an expandable, locking cap placed on the well riser. The area around the riser is dug out and a steel well vault or manhole cover placed over the riser and set almost flush to the ground.

to protect the well. The manhole cover should be water-tight and secured with bolts to prevent casual access. The well pad will then be constructed around the well vault and slightly mounded at the center and sloping away to prevent surface water from accumulating in the well vault.

- If completed as a stick-up well, the well riser is cut approximately 2.5 to 3 feet above the ground surface and an expandable, locking cap placed on the well riser. A steel guard pipe with hinged, locking cap is placed over the well riser as a protective casing. The bottom of the guard pipe will be set approximately 2 feet below ground surface and sealed by pouring concrete from the top of the annular grout around the pipe to grade. The concrete well pad should be completed at the same time. Weep holes will be drilled in the base of the guard pipe to facilitate draining of rainwater or purge water from inside the guard pipe.
- Bumper posts or bollards may be necessary for additional well protection, especially in high traffic areas. The bumper posts should be placed around the well pad in a configuration that provides maximum protection to the well and extend a minimum of 3 feet above the ground.

#### **7.4 Double Cased Wells**

Under certain site conditions, the use of a double-cased or telescoping (Type III) well may be necessary. Installation of double-cased wells may be required to prevent the interconnection of two separate aquifers, seal off a perched aquifer without creating a vertical hydraulic conduit, prevent cross-contamination during construction of wells in deeper aquifers hydro-stratigraphically below impacted aquifers, or case off highly impacted soils present above the aquifer to prevent potential “dragging down” of contaminants.

Similar to conventional wells, construction of double-cased wells can be accomplished using a variety of drilling methods. Well construction is initiated by “keying” a large diameter, outer casing into a stratigraphic zone of low permeability (clay layer or bedrock). The size of the outer casing should be a minimum of 2 inches greater than the outside diameter of the inner casing to allow installation of annular seal materials during well completion. A pilot borehole should be drilled through the overburden soil and/or contaminated zone into a clay confining layer or bedrock. The borehole for the outer casing should be of sufficient size to contain the outer casing with a minimum of 2 inches around the outside diameter to allow sufficient annular space for tremie or pressure grouting. The boring should extend a minimum of 2 feet into a clay layer and a minimum of 1 foot into bedrock, if possible, to ensure an adequate seal. The boring should never breach a confining layer or keyed zone under any circumstances.

Once the boring is completed, the outer casing can be set in the borehole and sealed with grout. The outer casing can be set two ways, with or without a bottom cap. If no bottom cap is applied, the casing is usually driven approximately 6 inches into the clay confining unit. A grout plug is generally placed in the bottom of the casing and once set, standing water in the casing is evacuated prior to drilling below the casing. As an alternative, a cap can be placed on the bottom of the casing and if set below the water table, the casing can be filled with clean, potable water to hold down the casing in the boring. Grouting should be conducted using tremie-grouting or pressure-grouting methods by pumping grout into the annular space between the outer casing and the borehole wall from the bottom of the casing to the ground surface. Grout around the casing should be allowed to cure at least 24 hours before attempting to drill through the bottom.

Once the grout is cured, a smaller diameter drill pipe/bit is used to bore through the grout plug or bottom cap to the desired well depth. The well is then constructed as described in Section 7.3 above.

#### **7.5 Post Installation Procedures**

- Wells should be permanently labelled or marked for identification. Well tags can be used to record the site name, well number, total depth, installation date, etc. At a minimum, the well number will be written in indelible marker or paint on both the outside of the protective casing and inside beneath the casing lid, as well as on the riser pipe.

- A measuring point will be marked on the top of the riser pipe for taking water level measurements. The measuring point can be notched using a knife or saw or can be marked with a waterproof marker or paint. The measuring point will also be the point which will be surveyed for vertical elevation data.
- Upon completion, the following measurements will be taken by the field geologist/engineer and recorded on the well construction diagram.
  - Depth to static water level
  - Depth of non-aqueous phase liquid (NAPL), if present
  - Total depth of well measured from top of casing (TOC)
  - Height of well casing above ground surface
  - Height of protective casing above ground surface
- All monitoring wells will be surveyed for horizontal and vertical control by a licensed surveyor.
- Investigation-derived waste (IDW) including drill cuttings, spent materials (e.g., PPE), and decontamination water should be properly managed in accordance with SOP 3-05, IDW Management.

## **8.0 Quality Control and Assurance**

- 8.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the SAP. Certain quality control (QC) measures should be taken to ensure proper well installation and construction in accordance with this SOP, project specific SAP, and applicable well standards.
- 8.2 The borehole will be checked for total open depth, and extended by further drilling or shortened by backfilling, as required before installation of the well materials.
- 8.3 Water level and NAPL presence will be checked during well installation to ensure that the positions of well screen, filter sand, and seals relative to water level conform to project requirements
- 8.4 The depth to top of each layer of annular materials (i.e., filter sand, bentonite, grout) will be verified and adjusted as necessary for proper placement.

## **9.0 Records, Data Analysis, Calculations**

All field information will be recorded in the field logbook and/or standardized field forms by field personnel. Field data recorded will include drilling contractor information, drilling methods, well material and construction information provided on the boring logs and well construction forms, observations or problems encountered during drilling, fluid level data, and any deviations from the procedures in this SOP and other project plans. Well Construction Forms (Attachment 1) will provide visual and descriptive information the monitoring well and are often the most critical form of documentation generated during the installation of a monitoring well. The field logbook is kept as a general log of activities and should not be used in place of the boring log.


## **10.0 Attachments or References**

- 10.1 Attachment 1 – Monitoring Well Construction Form

- 10.2 Environmental Protection Agency, United States (EPA). 1987. *A Compendium of Superfund Field Operations Methods*. Office of Solid Waste and Emergency Response. EPA/540/P-87/001.
- 10.3 EPA. 1990. *Handbook of Suggested Practices for the Design and Installation of Groundwater Monitoring Wells*. EPA/600/4-89/034. Office of Research and Development, Washington. March.
- 10.4 EPA. 1992. *RCRA Groundwater Monitoring Draft Technical Guidance*. EPA/530/R-93/001. Office of Solid Waste. November.
- 10.5 EPA, 2008. SESD Operating Procedure SESDGUID-101-R0: *Design and Installation of Monitoring Wells*. USEPA, Science and Ecosystem Support Division (SESD), Athens, Georgia. Effective Date February 18, 2008.
- 10.6 U.S. Army Corps of Engineers. 2008. Manual No. EM 385-1-1. *Safety and Health Requirements*. 15 November 2008. [http://140.194.76.129/publications/eng-manuals/em385-1-1/2008\\_English/toc.html](http://140.194.76.129/publications/eng-manuals/em385-1-1/2008_English/toc.html).
- 10.7 SOP 3-01, *Utility Clearance*.
- 10.8 SOP 3-05, *IDW Management*
- 10.9 SOP 3-06, *Equipment Decontamination*.
- 10.10 SOP 3-16, *Soil and Rock Classification*.

<i>Author</i>	<i>Reviewer</i>	<i>Revisions (Technical or Editorial)</i>
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)

# Attachment 1 Monitoring Well Construction Form

	Client: _____		<b>WELL ID:</b>	
	Project Number: _____			
	Site Location: _____		Date installed: _____	
	Well Location: _____ Coords: _____		Inspector: _____	
	Method: _____		Contractor: _____	

MONITORING WELL CONSTRUCTION DETAIL				
			Depth from G.S. (feet)	Elevation (feet)
				Datum _____
Measuring Point for Surveying & Water Levels	Top of Steel Guard Pipe		_____	_____
	Top of Riser Pipe		_____	_____
	Ground Surface (G.S.)		0.0	_____
Cement, Bentonite, Bentonite Slurry Grout, or Native Materials	Riser Pipe:			
	Length _____			
	Inside Diameter (ID) _____			
	Type of Material _____			
Bottom of Steel Guard Pipe				
% Cement _____	Top of Bentonite		_____	_____
% Bentonite _____	Bentonite Seal Thickness _____		_____	_____
% Native Materials _____	Top of Sand		_____	_____
	Top of Screen		_____	_____
	▼ Stabilized Water Level		_____	_____
	Screen:			
	Length _____			
	Inside Diameter (ID) _____			
	Slot Size _____			
	Type of Material _____			
	Type/Size of sand _____			
	Sand Pack Thickness _____			
	Bottom of Screen		_____	_____
	Bottom of Tail Pipe:		_____	_____
	Length _____			
	Bottom of Borehole		_____	_____
	Borehole Diameter _____			
Approved: _____				
Signature _____				
Date _____				
Describe Measuring Point: _____				



# Monitoring Well Development

## Procedure 3-13

### 1.0 Purpose and Scope

- 1.1 This standard operating procedure (SOP) describes the procedures used for developing newly installed monitoring wells and/or redeveloping existing wells.
- 1.2 The purpose of well development is to remove interferences from a well to provide better connection between the well and the formation, to improve pumping performance of the well, and to be able to collect more representative information from the well (e.g., samples, test results, etc.). Proper well development will:
- Remove drilling residuals (e.g., water, mud) from the borehole and surrounding formations;
  - Improve or restore hydraulic conductivity of the surrounding formations which may have been disturbed during the drilling process;
  - Remove residual fines from the well screen and sand pack (filter pack) materials, thus reducing turbidity of groundwater and permitting the collection of more representative groundwater samples.
- 1.3 There may be circumstances where well development is not desirable, for example, in the presence of non-aqueous phase liquids (NAPL) or other significant contamination if development could worsen the contaminant impact. If NAPL begins to intrude during development, the development process will be halted. This situation will be considered a cause for sample modification requiring approval by the CTO Manager and other stakeholders, as applicable.
- 1.4 The applicable well development procedures for a particular site may be subject to State or local regulatory requirements. In all cases, the project team should consult their local regulatory requirements and document the selected well development procedure in the project-specific Sampling and Analysis Plan (SAP). For project-specific information refer to the SAP, which takes precedence over these procedures.
- 1.5 This procedure is the Program-approved professional guidance for work performed by Resolution Consultants under the Comprehensive Long-Term Environmental Action Navy (CLEAN) contract (Contract Number N62470-11-D-8013).
- 1.6 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

### 2.0 Safety

- 2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the Contract Task Order (CTO) SAP and/or direction from the Site Safety Officer (SSO).
- 2.2 Monitoring well development may involve chemical hazards associated with potential contaminants in the soil or aquifer being characterized and may involve physical hazards associated with use of well development equipment.

### 3.0 Terms and Definitions

None.

## **4.0 Interferences**

- 4.1 Equipment/materials used for development may react with the groundwater during development. Appropriate development equipment has been selected for the anticipated condition of the groundwater.
- 4.2 Appropriate development methods such as using a surge-block to flush suspended fines in the groundwater in and out of the well screen can improve the yield of wells and improve their potential to be developed successfully. However, the effectiveness of development can be significantly reduced in wells that do not yield sufficient water to allow this flushing to take place.
- 4.3 For formations with a significant content of fine-grained materials (silts and clays), or wells with improperly sized screens, it may not be possible to reduce turbidity to commonly acceptable levels. Possible solutions may include collecting a sample even if excessively turbid, or installing a replacement well.
- 4.4 Development itself disturbs the surrounding formation and disrupts equilibrium conditions within the well. Groundwater samples will not be collected until a minimum of 24 hours after a well is developed to allow conditions to stabilize. For sites with fine-grained formations (silts and clays) and highly sorptive contamination, a longer time period between development and sampling should be considered.

## **5.0 Training and Qualifications**

### **5.1 Qualifications and Training**

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

### **5.2 Responsibilities**

- 5.2.1 The **CTO Manager** is responsible for ensuring that well development activities comply with this procedure. The **CTO Manager** is responsible for ensuring that all personnel involved in well development shall have the appropriate education, experience, and training to perform their assigned tasks.
- 5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.
- 5.2.3 The **Field Manager** is responsible for ensuring that all well development activities are conducted according to either this procedure or the applicable procedure presented in the project-specific SAP.
- 5.2.4 **Field sampling personnel** are responsible for the implementation of this procedure.
- 5.2.5 The field sampler and/or task manager is responsible for directly supervising the well development procedures to ensure that they are conducted according to this procedure and for recording all pertinent data collected during sampling.

## **6.0 Equipment and Supplies**

- 6.1 This equipment list was developed to aid in field organization and should be used in planning and preparation. Depending on the site-specific requirements and the development method selected, additional or alternative material and equipment may be necessary. In addition, for sites where groundwater is expected to be contaminated, the materials to be placed down the well and in contact with groundwater should be evaluated so that they are compatible with the chemical conditions expected in the well.
- 6.2 Equipment and materials used for well development may include, but is not limited to:

### **Well development equipment**

- Surge block

- Disposable Teflon bailers, appropriate to the diameter of the well(s): 1-inch to 1.5-inch for 2-inch inside diameter (ID) monitoring wells.
- Watterra® footvalve
- Electric submersible pump
- 12-volt power source for electric pump
- High density polyethylene (HDPE) tubing appropriately sized for Watterra® footvalve and/or electric submersible pump
- Drums or containers for storage of purge water
- Nephelometer to measure turbidity
- Multi-parameter water quality meter(s) to measure temperature, pH, conductivity, dissolved oxygen (DO), oxidation reduction potential (ORP)
- Instrument calibration solutions
- Water level meter
- Oil/water interface probe

#### **General equipment**

- Project-specific plans including the site-specific HASP and SAP
- Field notebook/field forms/site maps
- Indelible markers/pens
- 5-gallon buckets

#### **Equipment decontamination supplies** (refer to SOP 3-06, Equipment Decontamination)

- Health and safety supplies, including personal protective equipment (PPE)
- Appropriate hand tools
- Keys or combinations to access monitoring wells
- Distilled/deionized water supply
- Disposable bailer string (polypropylene)
- Plastic trash bags

## **7.0 Procedure**

Development generally consists of removing water and entrained sediment from the well until the water is clear (to the extent feasible) and the turbidity is reduced, which indicates the well is in good hydraulic connection with the surrounding formation. In addition to simply removing water, development can be improved when flushing through the well screen and gravel pack takes place in both directions, that is, both into the well and into the formation. This action breaks down sediment bridges that can occur in the formation or sand pack, which reduce the connection between the well and the formation

### **7.1 General Preparation**

- All down-well equipment should be decontaminated prior to use and between well locations in accordance with SOP 3-06, Equipment Decontamination
- Although equipment is decontaminated between well locations, if wells are known or suspected to be contaminated based on observations during well installation, it is recommended that well development be conducted in order from the least contaminated to the most contaminated well to minimize the chances of cross-contamination.
- Management of investigation-derived waste (IDW), including development purge water and miscellaneous expendable materials generated during the development process, will be conducted in accordance with SOP 3-05, IDW Management.

- Prior to accessing the well, the wellhead should be cleared of debris and/or standing water. Nothing from the ground surface should be allowed to enter the well.
- The depth to water and total well depth should be measured with a water level meter and recorded in the field logbook or on a Well Development Record (Attachment 1). This information will be used to calculate the volume of standing water (i.e., the well volume) within the well, and plan the specific details of the well development. If wells are suspected to contain NAPL, an oil/water interface probe should be used to measure liquid levels and depth to bottom of the well.
- Permanent monitoring wells will be developed no sooner than 24 hours after well installation is completed in order to allow well completion materials to set properly.

## 7.2 Monitoring Well Development Procedures

Generally, development will begin by gently surging the well with a surge block or bailer as described in Sections 7.2.1 and 7.2.2, respectively. Surging can become more vigorous as development progresses but initially the well must be gently surged to allow material blocking the screen to become suspended without damaging the well. Next, a bailer can be used to remove the sediment settled at the base of the well. A bailer, Watterra<sup>®</sup> pump, or electric submersible pump will then be used to purge the well, per Sections 7.2.2, 7.2.3, or 7.2.4, respectively. The well will be purged until the removed water becomes less turbid or per the requirements of the project-specific SAP, or State or local requirements. At this point the well will be surged again with a surge block or bailer. The well can be surged more vigorously at this point. After surging, the well will be purged again until the turbidity once again decreases. The surge/purge cycle should be completed at least three times during the development process. After the last surge, the well will be purged until the development completion criteria outlined in 7.3.2 or per the project-specific SAP are met.

### 7.2.1 Surge Block

The default method of well development is the use of a surge block in conjunction with pumping or bailing to remove sediment-laden water.

- The construction of the surge block must be appropriate for the diameter of the well. The surge block must be mounted on rods or other stiff materials to extend it to the appropriate depths and to allow for the surge block to be moved up and down in the well.
- Insert the surge block into the well and lower it slowly to the screened or open interval below the static water level. Start the surge action by slowly and gently moving the surge block up and down in the well. A slow initial surging, using plunger strokes of approximately 1 meter or 3 feet, will allow material which is blocking the screen to separate and become suspended.
- After 5 to 10 plunger strokes, remove water from the well using a separate bailer (Section 7.2.2) or pumping techniques (Sections 7.2.3 or 7.2.4). The returned water should be heavily laden with suspended fines. The water will be discharged to 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- In some cases, the bailer or Watterra<sup>®</sup> foot valve can act as a surge block, flushing water in and out of the well screen as groundwater is removed.
- Repeat the process of surging and pumping/bailing. As development continues, slowly increase the depth of surging to the bottom of the well screen. Surging within the riser portion of the well is neither necessary nor effective.

### 7.2.2 Bailer

- Tie a string or other cable securely to the bailer. Lower it to the screened or open interval of the monitoring well below the static water level.
- The bailer may be raised and lowered repeatedly within the screened interval to attempt to simulate the action of a surge block by pulling fines through the well screen, and pushing water out into the formation to break down bridging.

- With the bailer full of water, remove it from the well and discharge the water into 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- The Watterra® system (Section 7.2.3) or electric submersible pump (Section 7.2.4) may be used as a complementary development method to the bailer, especially when removal of additional water at a faster rate is beneficial.
- Continue alternately surging and bailing, monitoring the purge water periodically (Section 7.3.1) until development completion criteria are met (Section 7.3.2).

#### 7.2.3 Watterra® system

- Attach high-density polyethylene (HDPE) tubing to the decontaminated Watterra® pump foot valve
- Lower the foot valve and tubing assembly near the bottom of the well.
- Lift and lower the tubing to allow water to enter the Watterra® foot valve and travel up the tubing and discharge the water into 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- The lifting and lowering action of the Watterra® system will cause some surging action to aid in breaking up fine material in the surrounding formation.
- A bailer (Section 7.2.2) may be used as a complementary development method to the Watterra® system, especially during the initial stages of development when a high volume of sediment may be required to be removed.
- An electric submersible pump (Section 7.2.4) may also be used as a complementary development method to the Watterra® system, especially when more volume of water is desired to be pumped or the turbidity criteria cannot be met due to the surging action of the Watterra® system.
- Continue alternately surging and pumping, monitoring the purge water periodically (Section 7.3.1) until well development completion criteria are met (Section 7.3.2).

#### 7.2.4 Electric Submersible Pump

- Attach HDPE tubing to the decontaminated electric submersible pump.
- Lower the pump and tubing assembly near the bottom of the well, at least a few inches above the well total depth.
- Begin pumping, discharging the water into 5-gallon buckets or 55-gallon drums to be managed per the requirements presented in the project-specific SAP.
- Continue alternately surging and pumping, monitoring the purge water discharge periodically (Section 7.3.1) until well development completion criteria are met (Section 7.3.2).

### 7.3 Discharge Monitoring

#### 7.3.1 Monitoring the Progress of Development

The progress of the development is evaluated through visual observation of the suspended sediment load and measurement of the turbidity and other parameters in the purged discharge water. As development progresses, the water should become clearer, measured turbidity should decrease, and specific capacity (pumping rate divided by drawdown) should stabilize. Water quality parameters, including DO, conductivity, ORP, pH, temperature, and turbidity may be measured and recorded periodically to determine the progress of development using the criteria outlined in Section 7.3.2 or per the project-specific SAP. Water quality parameters should be measured on each well volume removed.

#### 7.3.2 Completion of Development

The well will be considered developed when the following criteria are met or per the criteria set forth in the project-specific SAP:

- A minimum of three times the standing water volume in a well (to include the well screen and casing plus saturated annulus, assuming 30 percent porosity) is removed.

- Groundwater parameters for three consecutive standing water volumes are within the following:
  - pH – within  $\pm 0.2$  units
  - Specific conductivity – within  $\pm 3\%$
  - ORP – within  $\pm 10$  mV
  - Temperature – within  $\pm 1$  degree Celsius
  - Turbidity – at or below 10 nephelometric turbidity units (NTU) or within  $\pm 10\%$  if above 10 NTU.
- The sediment thickness remaining within the well is less than 1 percent of the screen length or less than 30 millimeters (0.1 ft) for screens equal to or less than 10 feet long.

Dissolved oxygen (DO) readings may be recorded but DO readings will not be used as development completion criteria because DO may not stabilize.

If the well has slow groundwater recharge and is purged dry, the well will be considered developed when bailed or pumped dry three times in succession and the turbidity has decreased, or per the requirements set forth in the project-specific SAP. Water quality parameters may be recorded if feasible using the flow-through cell.

If any water is added to the well's borehole during development or drilling, three times the volume of water added will also be removed during well development, or per the requirements set forth in the project-specific SAP.

#### 7.4 Development of Wells with Low Yield

Water is the primary mechanism to remove fines and flush water through the gravel pack for effective development. Therefore, development can be a challenge in wells that do not yield sufficient water to recharge when water is removed. However, often these wells are the most in need of development to improve their performance as they are typically installed in low permeability formations with a high content of fines. Development of these wells can improve their yield.

The surging portion of the development can be successfully performed in a well with standing water regardless of its yield. It is the subsequent removal of fine materials that is hindered when insufficient water is recharged to the well. When wells go dry or drawdown significantly during development, development can be performed intermittently, allowing sufficient water to recharge prior conducting the next stage of surging. These intermittent procedures can take place hours or even days apart, depending on project-specific time constraints.

#### 7.5 Wells containing NAPL

Additional care should be taken when planning development of wells that contain NAPL. If the NAPL is flammable, there are health and safety as well as handling issues to consider. If NAPL in excess of a persistent sheen is noted, the recharge rate will be evaluated through hand bailing. In most cases, it is generally preferable to remove NAPL by bailing to the extent practical prior to performing development. Groundwater parameters, excluding turbidity, will not be collected during well development if NAPL or excessive sheen is noticed in the purged water during development to ensure the meter probes are not fouled or destroyed. Well development will be halted.

Development by surging or pumping the well dry can result in the spreading of NAPL vertically in the soil column around the well. These methods can be used, if information exists describing the vertical thickness of the NAPL smear zone around the well, and if the methods do not result in mounding or drawdown that exceeds this thickness. Alternate methods such as bailing may also be used, but any method should not allow the well to be pumped dry or result in significant drawdown that would spread the NAPL vertically.



## 7.6 Temporary Well Points

For certain projects, temporary well points (TWPs) may be installed to collect groundwater samples at a site. Since no sand pack, bentonite chips, or bentonite grout are generally used in the construction of the TWPs, development can proceed as soon as sufficient water has entered the well to static conditions. Due to the small diameter of these wells, generally ¾-inch to 1-inch ID, development will be performed using either a small diameter (0.5-inch) bailer and/or a peristaltic pump with dedicated tubing. The TWPs will have minimal water column and may purge dry during development. However, attempts will be made to remove fines from the well prior to sampling. Purging and sampling may occur as soon as approximately 80% of the static water has re-entered the TWP, or per the requirements set forth in the project-specific SAP.

## 8.0 Quality Control and Assurance

- 8.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the project-specific SAP.
- 8.2 Quality control (QC) requirements are dependent on project-specific sampling objectives. The project-specific SAP will provide requirements for equipment decontamination (frequency and materials) and IDW handling.

## 9.0 Records, Data Analysis, Calculations

- 9.1 All data and information (e.g., development method used) must be documented on field data sheets (Attachment 1) or within site logbooks with permanent ink. Data recorded may include the following:
- Well Location
  - Weather conditions
  - Date and Time
  - Purge Method
  - Reading/measurements obtained

## 10.0 Attachments or References

Attachment 1 – Well Development Record

SOP 3-05, *IDW Management*.

SOP 3-06, *Equipment Decontamination*.

<i>Author</i>	<i>Reviewer</i>	<i>Revisions (Technical or Editorial)</i>
Shawn Dolan Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (June 2012)

## Attachment 1 Well Development Record



### Well/Piezometer Development Record

Well ID:

Client: \_\_\_\_\_

Project No: \_\_\_\_\_ Date: \_\_\_\_\_ Developer: \_\_\_\_\_

Site Location: \_\_\_\_\_

#### Well/Piezometer Data

Well ☐ Piezometer ☐ Diameter \_\_\_\_\_ Material \_\_\_\_\_

Measuring Point Description \_\_\_\_\_ Geology at Screen Interval (if known) \_\_\_\_\_

Depth to Top of Screen (ft.) \_\_\_\_\_

Depth to Bottom of Screen (ft.) \_\_\_\_\_ Time of Water Level Measurement \_\_\_\_\_

Total Well Depth (ft.) \_\_\_\_\_ Calculate Purge Volume (gal.) \_\_\_\_\_

Depth to Static Water Level (ft.) \_\_\_\_\_ Disposal Method \_\_\_\_\_

Headspace \_\_\_\_\_

Original Well Development ☐ Redevelopment ☐ Date of Original Development \_\_\_\_\_

#### DEVELOPMENT METHOD

#### PURGE METHOD

Time	Total Volume Purged (gal.)	Flow Rate (gpm)	Turbidity (NTU)	Color	pH	Temp	Other

#### ACCEPTANCE CRITERIA (from workplan)

Minimum Purge Volume Required \_\_\_\_\_ gallons

Maximum Turbidity Allowed \_\_\_\_\_ NTUs

Stabilization of parameters \_\_\_\_\_ %

Has required volume been removed

Has required turbidity been reached

Has parameters stabilized

If no or N/A explain below:

Yes No N/A

☐ ☐ ☐

☐ ☐ ☐

☐ ☐ ☐

Signature \_\_\_\_\_

Date: \_\_\_\_\_

# Monitoring Well Sampling

## Procedure 3-14

### 1.0 Purpose and Scope

- 1.1 This standard operating procedure (SOP) describes the actions to be used during monitoring well sampling activities and establishes the method for sampling groundwater monitoring wells for water-borne contaminants and general groundwater chemistry. The objective is to obtain groundwater samples that are representative of aquifer conditions with as little alteration to water chemistry as possible.
- 1.2 This procedure is the Program-approved professional guidance for work performed by Resolution Consultants under the Comprehensive Long-Term Environmental Action Navy (CLEAN) contract (Contract Number N62470-11-D-8013).
- 1.3 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

### 2.0 Safety

- 2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to sampling the first well. All field sampling personnel responsible for sampling activities must review the project-specific health and safety plan (HASP) paying particular attention to the control measures planned for the well sampling tasks. Conduct preliminary area monitoring of sampling wells to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor phase and liquid matrix through the use of appropriate personal protective equipment (PPE).
- 2.2 Observe standard health and safety practices according to the project-specific HASP. Suggested minimum protection during well sampling activities includes inner disposable vinyl gloves, outer chemical-protective nitrile gloves and rubberized steel-toed boots. Half-face respirators and cartridges and Tyvek® suits may be necessary depending on the contaminant concentrations. Refer to the project-specific HASP for the required PPE.
- 2.3 Physical Hazards associated with Well Sampling
  - To avoid lifting injuries associated with pump and bailers retrieval, use the large muscles of the legs, not the back.
  - Stay clear of all moving equipment, and avoid wearing loose fitting clothing.
  - When using tools for cutting purposes, cut away from yourself. The use of appropriate, task specific cutting tools is recommended.
  - To avoid slip/trip/fall conditions as a result of pump discharge, use textured boots/boot cover bottoms.
  - To avoid heat/cold stress as a result of exposure to extreme temperatures and PPE, drink electrolyte replacement fluids (1 to 2 cups per hour is recommended) and, in cases of extreme cold, wear fitted insulating clothing.
  - Be aware of restricted mobility due to PPE.

### **3.0 Terms and Definitions**

None.

### **4.0 Interferences**

4.1 Potential interferences could result from cross-contamination between samples or sample locations. Minimization of the cross-contamination will occur through the following:

- The use of clean sampling tools at each location as necessary.
- Avoidance of material that is not representative of the media to be sampled.

### **5.0 Training and Qualifications**

#### **5.1 Qualifications and Training**

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

#### **5.2 Responsibilities**

5.2.1 The **Contract Task Order (CTO) Manager** is responsible for ensuring that monitoring well sampling activities comply with this procedure. The **CTO Manager** is responsible for ensuring that all field sampling personnel involved in monitoring well sampling shall have the appropriate education, experience, and training to perform their assigned tasks.

5.2.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.

5.2.3 The **Field Manager** is responsible for ensuring that all field sampling personnel follow these procedures.

5.2.4 **Field sampling personnel** are responsible for the implementation of this procedure.

5.2.5 The field sampler and/or task manager is responsible for directly supervising the groundwater sampling procedures to ensure that they are conducted according to this procedure and for recording all pertinent data collected during sampling.

### **6.0 Equipment and Supplies**

#### **6.1 Purging and Sampling Equipment**

- Pump (Peristaltic, Portable Bladder, Submersible)
- Polyethylene or Teflon bladders (for portable bladder pumps)
- Bladder pump controller (for portable bladder pumps)
- Air compressor (for portable bladder pumps)
- Nitrogen cylinders (for portable bladder pumps)
- 12-volt power source
- Polyethylene inlet and discharge tubing (except for VOC analysis which requires Teflon tubing)
- Silicone tubing appropriate for peristaltic pump head
- Teflon bailer appropriately sized for well

- Disposable bailer string (polypropylene)
- Individual or multi-parameter water quality meter(s) with flow-through cell to measure temperature, pH, specific conductance, dissolved oxygen (DO), oxidation reduction potential (ORP), and/or turbidity
- Turbidity meter
- Water level meter
- Oil/water interface probe

## 6.2 General Equipment

- Sample kit (i.e., bottles, labels, preservatives, custody records and tape, cooler, ice)
- Sample Chain-of-Custody (COC) forms
- Sample Collection Records
- Sample packaging and shipping supplies
- Waterproof marker or paint
- Distilled/deionized water supply
- Water dispenser bottles
- Flow measurement cup or bucket
- 5-gallon buckets
- Instrument calibration solutions
- Stopwatch or watch
- Disposable Nitrile gloves
- Paper towels
- Trash bags
- Zipper-lock bags
- Equipment decontamination supplies
- Health and safety supplies (as required by the HASP)
- Approved plans such as: project-specific HASP and Sampling and Analysis Plan (SAP)
- Well keys or combinations
- Monitoring well location map(s)
- Field project logbook/pen

## 7.0 Calibration or Standardization

- 7.1 Field instruments will be calibrated daily according to the requirements of the SAP and manufacturer's specifications for each piece of equipment. Equipment will be checked daily with the calibration solutions at the end of use of the equipment. Calibration records shall be recorded in the field logbook or appropriate field form.
- 7.2 If readings are suspected to be inaccurate, the equipment shall be checked with the calibration solutions and/or re-calibrated.

## **8.0 Procedure**

### **8.1 Preparation**

#### **8.1.1 Site Background Information**

Establish a thorough understanding of the purposes of the sampling event prior to field activities. Conduct a review of all available data obtained from the site and pertinent to the water sampling. Review well history data including, but not limited to, well locations, sampling history, purging rates, turbidity problems, previously used purging methods, well installation methods, well completion records, well development methods, previous analytical results, presence of an immiscible phase, historical water levels, and general hydrogeologic conditions.

Previous groundwater development and sampling logs give a good indication of well purging rates and the types of problems that might be encountered during sampling, such as excessive turbidity and low well yield. They may also indicate where dedicated pumps are placed in the water column. To help minimize the potential for cross-contamination, well purging and sampling and water level measurement collection shall proceed from the least contaminated to the most contaminated well as indicated by previous analytical results. This order may be changed in the field if conditions warrant it, particularly if dedicated sampling equipment is used. A review of prior sampling procedures and results may also identify which purging and sampling techniques are appropriate for the parameters to be tested under a given set of field conditions.

#### **8.1.2 Groundwater Analysis Selection**

Establish the requisite field and laboratory analyses prior to water sampling. Decide on the types and numbers of quality assurance/quality control (QA/QC) samples to be collected (refer to the project-specific SAP), as well as the type and volume of sample preservatives, the type and number of sample containers, the number of coolers required, and the quantity of ice or other chilling materials. The field sampling personnel shall ensure that the appropriate number and size sample containers are brought to the site, including extras in case of breakage or unexpected field conditions. Refer to the project-specific SAP for the project analytical requirements.

### **8.2 Groundwater Sampling Procedures**

Groundwater sampling procedures at a site shall include:

- 1) An evaluation of the well security and condition prior to sampling;
- 2) Decontamination of equipment;
- 3) Measurement of well depth to groundwater;
- 4) Assessment of the presence or absence of an immiscible phase;
- 5) Assessment of purge parameter stabilization;
- 6) Purging of static water within the well and well bore; and
- 7) Obtaining a groundwater sample.

Each step is discussed in sequence below. Depending upon specific field conditions, additional steps may be necessary. As a rule, at least 24 hours should separate well development and well sampling events. In all cases, consult the State and local regulations for the site, which may require more stringent time separation between well development and sampling.



### 8.2.1 Well Security and Condition

At each monitoring well location, observe the conditions of the well and surrounding area. The following information may be noted on a Groundwater Sample Collection Record (Attachment 1) or in the field logbook:

- Condition of the well's identification marker.
- Condition of the well lock and associated locking cap.
- Integrity of the well – well pad condition, protective outer casing, obstructions or kinks in the well casing, presence of water in the annular space, and the top of the interior casing.
- Condition of the general area surrounding the well.

### 8.2.2 Decontamination of Equipment

Where possible, dedicated supplies should be used at each well location to minimize the potential for cross-contamination and minimize the amount of investigation derived waste (IDW) fluids resulting from the decontamination process. If decontamination is necessary, establish a decontamination station before beginning sampling. The station shall consist of an area of at least 4 feet by 2 feet covered with plastic sheeting and be located upwind of the well being sampled. The station shall be large enough to fit the appropriate number of wash and rinse buckets, and have sufficient room to place equipment after decontamination. One central cleaning area may be used throughout the entire sampling event. The area around the well being sampled shall also be covered with plastic sheeting to prevent spillage. Further details are presented in SOP 3-06, Equipment Decontamination.

Decontaminate each piece of equipment prior to entering the well. Also, conduct decontamination prior to sampling at a site, even if the equipment has been decontaminated subsequent to its last usage. Additionally, decontaminate each piece of equipment used at the site prior to leaving the site. It is only necessary to decontaminate dedicated sampling equipment prior to installation within the well. Do not place clean sampling equipment directly on the ground or other contaminated surfaces prior to insertion into the well. Dedicated sampling equipment that has been certified by the manufacturer as being decontaminated can be placed in the well without on-site decontamination.

### 8.2.3 Measurement of Static Water Level Elevation

Before purging the well, measure water levels in all of the wells within the zone of influence of the well being purged. The best practice, if possible, is to measure all site wells (or wells within the monitoring well network) prior to sampling. If the well cap is not vented, remove the cap several minutes before measurement to allow water levels to equilibrate to atmospheric pressure.

Measure the depth to standing water and the total depth of the well to the nearest 0.01 foot to provide baseline hydrologic data, to calculate the volume of water in the well, and to provide information on the integrity of the well (e.g., identification of siltation problems). If not already present, mark an easily identified reference point for water level measurements which will become the measuring point for all water level measurements. This location and elevation must be surveyed.

The device used to measure the water level surface and depth of the well shall be sufficiently sensitive and accurate in order to obtain a measurement to the nearest 0.01 foot reliably. An electronic water level meter will usually be appropriate for this measurement; however, when the groundwater within a particular well is highly contaminated, an inexpensive weighted tape measure can be used to determine well depth to prevent adsorption of contaminants onto the meter tape. The presence of light, non-aqueous phase liquids (LNAPLs) and/or dense, non-aqueous phase liquids (DNAPLs) in a well requires measurement of the elevation of the top and the bottom of the product, generally using an interface probe. Water levels in such wells must then be corrected for density effects to accurately determine the elevation of the water table.

At each location, measure water levels several times in quick succession to ensure that the well has equilibrated to atmospheric conditions prior to recording the measurement. As stated above, measure all site wells (or wells within the monitoring well network) prior to sampling whenever possible. This will provide a water level database that describes water levels across the site at one time (a synoptic sampling). Prior to sampling, measure the water level in each well immediately prior to purging the well to ascertain that static conditions have been achieved prior to sampling.

#### 8.2.4 Detection of Immiscible Phase Layers

Complete the following steps for detecting the presence of LNAPL and DNAPL before the well is purged for conventional sampling. These procedures may not be required for all wells. Consult the project-specific SAP to determine if assessing the presence of LNAPL and/or DNAPL is necessary.

- 1) Sample the headspace in the wellhead immediately after the well is opened for organic vapors using either a PID or an organic vapor analyzer, and record the measurements.
- 2) Lower an interface probe into the well to determine the existence of any immiscible layer(s), LNAPL and/or DNAPL, and record the measurements.
- 3) Confirm the presence or absence of an immiscible phase by slowly lowering a clear bailer to the appropriate depth, then visually observing the results after sample recovery.
- 4) In rare instances, such as when very viscous product is present, it may be necessary to utilize hydrocarbon- and water-sensitive pastes for measurement of LNAPL thickness. This is accomplished by smearing adjacent, thin layers of both hydrocarbon- and water-sensitive pastes along a steel measuring tape and inserting the tape into the well. An engineering tape showing tenths and hundredths of feet is required. Record depth to water, as shown by the mark on the water-sensitive paste, and depth to product, as shown by the mark on the product-sensitive paste. In wells where the approximate depth to water and product thickness are not known, it is best to apply both pastes to the tape over a fairly long interval (5 feet or more). Under these conditions, measurements are obtained by trial and error and may require several insertions and retrievals of the tape before the paste-covered interval of the tape encounters product and water. In wells where approximate depths of air-product and product-water interfaces are known, pastes may be applied over shorter intervals. Water depth measurements should not be used in preparation of water table contour maps until they are corrected for depression by the product.
- 5) If the well contains an immiscible phase, it may be desirable to sample this phase separately. Section 8.2.6 presents immiscible phase sampling procedures. It may not be meaningful to conduct water sample analysis of water obtained from a well containing LNAPLs or DNAPLs. Consult the **CTO Manager** and **Program Quality Manager** if this situation is encountered.

#### 8.2.5 Purging Equipment and Use

##### **General Requirements**

The water present in a well prior to sampling may not be representative of in situ groundwater quality and shall be removed prior to sampling. Handle all groundwater removed from potentially contaminated wells in accordance with the IDW handling procedures in SOP 3-05, IDW Management. Purging shall be accomplished by methods as indicated in the project-specific SAP or by those required by State requirements. For the purposes of this SOP, purging methods will be described by removing groundwater from the well using low-flow techniques.

According to the U.S. Environmental Protection Agency (EPA) (EPA, 1996), the rate at which groundwater is removed from the well during purging ideally should be less than 0.2 to 0.3 liters/minute. EPA further states that wells should be purged at rates below those used to develop the well to prevent further development of the well, to prevent damage to the well, and to avoid disturbing accumulated

corrosion or reaction products in the well. EPA also indicates that wells should be purged at or below their recovery rate so that migration of water in the formation above the well screen does not occur.

Realistically, the purge rate should be low enough that substantial drawdown in the well does not occur during purging. In addition, a low purge rate will reduce the possibility of stripping volatile organic compounds (VOCs) from the water, and will reduce the likelihood of increasing the turbidity of the sample due to mobilizing colloids in the subsurface that are immobile under natural flow conditions.

The field sampler shall ensure that purging does not cause formation water to cascade down the sides of the well screen. Wells should not be purged to dryness if recharge causes the formation water to cascade down the sides of the screen, as this will cause an accelerated loss of volatiles. This problem should be anticipated based on the results of either the well development task or historical sampling events. In general, place the intake of the purge pump in the middle of the saturated screened interval within the well to allow purging and at the same time minimize disturbance/overdevelopment of the screened interval in the well. Water shall be purged from the well at a rate that does not cause recharge water to be excessively agitated unless an extremely slow recharging well is encountered where complete evacuation is unavoidable. During the well purging procedure, collect water level and/or product level measurements to assess the hydraulic effects of purging. Sample the well when it recovers sufficiently to provide enough water for the analytical parameters specified. If the well is purged dry, allow the well to recover sufficiently to provide enough water for the specified analytical parameters, and then sample it.

Evaluate water samples on a regular basis during well purging and analyze them in the field preferably using in-line devices (i.e., flow through cell) for temperature, pH, specific conductivity, dissolved oxygen (DO), and oxidation-reduction (redox) potential. Turbidity should be measured separately (outside of the flow-through cell) with a nephelometer or similar device.

Readings should be taken every 2 to 5 minutes during the purging process. These parameters are measured to demonstrate that the natural character of the formation waters has been restored.

Purging shall be considered complete per the requirements set forth in the project-specific SAP, State requirements, or when three consecutive field parameter measurements of temperature, pH, specific conductivity, DO and ORP stabilize within approximately 10 percent and the turbidity is at or below 10 nephelometric turbidity units (NTU) or within  $\pm 10\%$  if above 10 NTU. This criterion may not be applicable to temperature if a submersible pump is used during purging due to the heating of the water by the pump motor. Enter all information obtained during the purging and sampling process into a groundwater sampling log. Attachment 1 shows an example of a groundwater sampling log and the information typically included in the form. Whatever form is used, all blanks need to be completed on the field log during field sampling.

Groundwater removed during purging shall be stored according to the project-specific SAP or per SOP 3-05, IDW Management.

### **Purging Equipment and Methods**

#### ***Submersible Pump***

A stainless steel submersible pump may be utilized for purging both shallow and deep wells prior to sampling the groundwater for semivolatile and non-volatile constituents, but are generally not preferred for VOCs unless there are no other options (e.g., well over 200 feet deep). For wells over 200 feet deep, the submersible pump is one of the few technologies available to feasibly accomplish purging under any yield conditions. For shallow wells with low yields, submersible pumps are generally inappropriate due to overpumpage of the wells (<1 gallon per minute), which causes increased aeration of the water within the well.

Steam clean or otherwise decontaminate the pump and discharge tubing prior to placing the pump in the well. The submersible pump shall be equipped with an anti-backflow check valve to limit the amount of

water that will flow back down the drop pipe into the well. Place the pump in the middle of the saturated screened interval within the well and maintain it in that position during purging.

#### ***Bladder Pump***

A stainless steel bladder pump can be utilized for purging and sampling wells up to 200 feet in depth for volatile, semivolatile, and non-volatile constituents. Use of the bladder pump is most effective in low to moderate yield wells and are often the preferred method for low-flow sampling. When sampling for VOCs and/or SVOCs, Teflon bladders should be used. Polyethylene bladders may be used when sampling for inorganics.

Either compressed dry nitrogen or compressed dry air, depending upon availability, can operate the bladder pump. The driving gas utilized must be dry to avoid damage to the bladder pump control box. Decontaminate the bladder pump prior to use.

#### ***Centrifugal, Peristaltic, or Diaphragm Pump***

A centrifugal, peristaltic, or diaphragm pump may be utilized to purge a well if the water level is within 20 feet of ground surface. New or dedicated tubing is inserted into the midpoint of the saturated screened interval of the well. Water should be purged at a rate that satisfies low-flow requirements (i.e., does not cause drawdown). Centrifugal, peristaltic, or diaphragm pump are generally discouraged for VOCs sampling; however, follow methods allowed per the project-specific SAP or State requirements.

#### ***Air Lift Pump***

Airlift pumps are not appropriate for purging or sampling.

#### ***Bailer***

Avoid using a bailer to purge a well because it can result in overdevelopment of the well and create excessive purge rates. If a bailer must be used, the bailer should either be dedicated or disposable. Teflon-coated cable mounted on a reel is recommended for lowering the bailer in and out of the well.

Lower the bailer below the water level of the well with as little disturbance of the water as possible to minimize aeration of the water in the well. One way to gauge the depth of water on the reel is to mark the depth to water on the bailer wire with a stainless steel clip. In this manner, less time is spent trying to identify the water level in the well.

### **8.2.6 Monitoring Well Sampling Methodologies**

#### **Sampling Light, Non-Aqueous Phase Liquids (LNAPL)**

Collect LNAPL, if present, prior to any purging activities. The sampling device shall generally consist of a dedicated or disposable bailer equipped with a bottom-discharging device. Lower the bailer slowly until contact is made with the surface of the LNAPL, and to a depth less than that of the immiscible fluid/water interface depth as determined by measurement with the interface probe. Allow the bailer to fill with LNAPL and retrieve it.

When sampling LNAPLs, never drop bailers into a well and always remove them from the well in a manner that causes as little agitation of the sample as possible. For example, the bailer should not be removed in a jerky fashion or be allowed to continually bang against the well casing as it is raised. Teflon bailers should always be used when sampling LNAPL. The cable used to raise and lower the bailer shall be composed of an inert material (e.g., stainless steel) or coated with an inert material (e.g., Teflon).

#### **Sampling Dense, Non-Aqueous Phase Liquids (DNAPL)**

Collect DNAPL prior to any purging activities. The best method for collecting DNAPL is to use a double-check valve, stainless steel bailer, or a Kemmerer (discrete interval) sampler. The sample shall be collected by slow, controlled lowering of the bailer to the bottom of the well, activation of the closing device, and retrieval.

### **Groundwater Sampling Methodology**

The well shall be sampled when groundwater within it is representative of aquifer conditions per the methods described in Section 8.2.5. Prior to sampling the flow-through cell shall be removed and the samples collected directly from the purge tubing. Flow rates shall not be adjusted once aquifer conditions are met. Additionally, a period of no more than 2 hours shall elapse between purging and sampling to prevent groundwater interaction with the casing and atmosphere. This may not be possible with a slowly recharging well. Measure and record the water level prior to sampling in order to monitor drawdown when using low-flow techniques and gauge well volumes removed and recharged when using non-low-flow techniques.

Sampling equipment (e.g., especially bailers) shall never be dropped into the well, as this could cause aeration of the water upon impact. Additionally, the sampling methodology utilized shall allow for the collection of a groundwater sample in as undisturbed a condition as possible, minimizing the potential for volatilization or aeration. This includes minimizing agitation and aeration during transfer to sample containers, minimizing exposure to sunlight, and immediately placing the sample on ice once collected.

Sampling equipment shall be constructed of inert material. Equipment with neoprene fittings, polyvinyl chloride (PVC) bailers, Tygon® tubing, silicon rubber bladders, neoprene impellers, polyethylene, and Viton® are not acceptable when sampling for organics. If bailers are used, an inert cable/chain (e.g., fluorocarbon resin-coated wire or stainless steel wire or cable) shall be used to raise and lower the bailer. Dedicated equipment is highly recommended for all sampling programs.

#### ***Submersible Pumps***

The submersible pump must be specifically designed for groundwater sampling (i.e., pump composed of stainless steel and Teflon, sample discharge lines composed of Teflon) and must have a controller mechanism allowing the required low-flow rate. Adjust the pump rate so that flow is continuous and does not pulsate to avoid aeration and agitation within the sample discharge lines. Run the pump for several minutes at the low-flow rate used for sampling to ensure that the groundwater in the lines was obtained at the low-flow rate.

#### ***Bladder Pumps***

A gas-operated stainless steel bladder pump with adjustable flow control and equipped with a Teflon bladder and Teflon-lined tubing can be effectively utilized to collect a groundwater sample and is considered to be the best overall device for sampling inorganic and organic constituents. If only inorganics are being sampled, polyvinyl bladders and tubing may be used. Operate positive gas displacement bladder pumps in a continuous manner so that they minimize discharge pulsation that can aerate samples in the return tube or upon discharge.

When using a compressor, take several precautions. If the compressor is being powered by a gasoline generator, position the generator downwind of the well. Ground fault circuit interrupters (GFCIs) should always be used when using electric powered equipment. Do not connect the compression hose from the compressor to the pump controller until after the engine has been started.

When all precautions are completed and the compressor has been started, connect the compression hose to the pump controller. Slowly adjust the control knobs to discharge water in the shortest amount of time while maintaining a near constant flow. This does not mean that the compressor must be set to discharge the water as hard as possible. The optimal setting is one that produces the largest volume of purge water per minute (not per purge cycle) while maintaining a near constant flow rate.

Prior to sampling, adjust the flow rate (purge rate) to yield 100 to 300 mL/minute. Avoid settings that produce pulsating streams of water instead of a steady stream if possible. Operate the pump at this low flow rate for several minutes to ensure that drawdown is not occurring. At no time shall the sample flow rate exceed the flow rate used while purging.

For those samples requiring filtration, it is recommended to use an in-line high capacity filter after all non-filtered samples have been collected.

#### ***Peristaltic Pumps:***

A peristaltic pump is a type of positive displacement pump that moves water via the process of peristalsis. The pump uses a flexible hose fitted inside a circular pump casing. A rotor with cams compresses the flexible tube as the rotor turns, which forces the water to be pumped to move through the tube. In peristaltic pumps, no moving parts of the pump are in contact with the water being pumped. Displacement is determined by tube size, so delivery rate can only be changed during operation by varying pump speed. Peristaltic pumps are simple and quite inexpensive for the flow rates they provide.

There are several methods available for transferring the sample into the laboratory containers. The selected method may vary based on State requirements and should be documented in the project-specific SAP. Samples typically can be collected directly from the discharge end of the Teflon tubing, after it has been disconnected from the flow through cell. For volatile analyses, the sampler should make sure that the pump is set such that a smooth laminar flow is achieved. In all cases, the project team should consult their local regulatory requirements and document the selected sample collection procedure in the project-specific SAP.

#### ***Bailers***

A single- or double-check valve Teflon or stainless steel bailer equipped with a bottom discharging device can be utilized to collect groundwater samples. Bailers have a number of disadvantages, however, including a tendency to alter the chemistry of groundwater samples due to degassing, volatilization, and aeration; the possibility of creating high groundwater entrance velocities; differences in operator techniques resulting in variable samples; and difficulty in determining where in the water column the sample was collected. Therefore, use bailers for groundwater sampling only when other types of sampling devices cannot be utilized for technical, regulatory, or logistical reasons.

Dedicated or disposable bailers should always be used in order to eliminate the need for decontamination and to limit the potential of cross-contamination. Each time the bailer is lowered to the water table, lower it in such a way as to minimize disturbance and aeration of the water column within the well.

#### **8.2.7 Sample Handling and Preservation**

Many of the chemical constituents and physiochemical parameters to be measured or evaluated during groundwater monitoring programs are chemically unstable and require preservation. The U.S. EPA document entitled, *Test Methods for Evaluating Solid Waste – Physical/Chemical Methods (SW-846)* (EPA 1997), includes a discussion of appropriate sample preservation procedures. In addition, SW-846 provides guidance on the types of sample containers to use for each constituent or common set of parameters. In general, check with specific laboratory or State requirements prior to obtaining field samples. In many cases, the laboratory will supply the necessary sample bottles and required preservatives. In some cases, the field sampling personnel may add preservatives in the field.

Improper sample handling may alter the analytical results of the sample. Therefore, transfer samples in the field from the sampling equipment directly into the container that has been prepared specifically for that analysis or set of compatible parameters as described in the project-specific SAP. It is not an acceptable practice for samples to be composited in a common container in the field and then split in the laboratory, or poured first into a wide mouth container and then transferred into smaller containers.

Collect groundwater samples and place them in their proper containers in the order of decreasing volatility and increasing stability. A preferred collection order for some common groundwater parameters is:

1. VOCs and total organic halogens (TOX)



2. Dissolved gases, total organic carbon (TOC), total fuel hydrocarbons
3. Semivolatile organics, pesticides
4. Total metals, general minerals (unfiltered)
5. Dissolved metals, general minerals (filtered)
6. Phenols
7. Cyanide
8. Sulfate and chloride
9. Nitrate and ammonia
10. Radionuclides

When sampling for VOCs, collect water samples in vials or containers specifically designed to prevent loss of VOCs from the sample. The analytical laboratory performing the analysis shall provide these vials. Collect groundwater from the sampling device in vials by allowing the groundwater to slowly flow along the sides of the vial. Sampling equipment shall not touch the interior of the vial. Fill the vial above the top of the vial to form a positive meniscus with no overflow. No headspace shall be present in the sample container once the container has been capped. This can be checked by inverting the bottle once the sample is collected and tapping the side of the vial to dislodge air bubbles. Sometimes it is not possible to collect a sample without air bubbles, particularly water that has high concentrations of dissolved gasses. In these cases, the field sampling personnel shall document the occurrence in the field logbook and/or sampling worksheet at the time the sample was collected. Likewise, the analytical laboratory shall note in the laboratory analysis reports any headspace in the sample container(s) at the time of receipt by the laboratory.

### **Special Handling Considerations**

In general, samples for organic analyses should not be filtered. However, high turbidity samples for PCB analysis may require filtering. Consult the project-specific SAP for details on filtering requirements. Samples shall not be transferred from one container to another because this could cause aeration or a loss of organic material onto the walls of the container. TOX and TOC samples should be handled in the same manner as VOC samples.

When collecting total and dissolved metals samples, the samples should be collected sequentially. The total metals sample is collected from the pump unfiltered. The dissolved metals sample is collected after filtering with a 0.45-micron membrane in-line filter. Allow at least 500 mL of effluent to flow through the filter prior to sampling to ensure that the filter is thoroughly wetted and seated in the filter capsule. If required by the project-specific SAP, include a filter blank for each lot of filters used and always record the lot number of the filters.

### **Field Sampling Preservation**

Preserve samples immediately upon collection. Ideally, sampling containers will be pre-preserved with a known concentration and volume of preservative. Certain matrices that have alkaline pH (greater than 7) may require more preservative than is typically required. An early assessment of preservation techniques, such as the use of pH strips after initial preservation, may therefore be appropriate. Guidance for the preservation of environmental samples can be found in the U.S. EPA *Handbook for Sampling and Sample Preservation of Water and Wastewater* (EPA 1982). Additional guidance can be found in other U.S. EPA documents (EPA 1992, 1996).

### **Field Sampling Log**

A groundwater sampling log provided as Attachment 1 shall document the following:

- Identification of well

- Well depth
- Static water level depth and measurement technique
- Presence of immiscible layers and detection method
- Well yield
- Purge volume and pumping rate
- Time that the well was purged
- Sample identification numbers
- Well evacuation procedure/equipment
- Sample withdrawal procedure/equipment
- Date and time of collection
- Types of sample containers used
- Preservative(s) used
- Parameters requested for analysis
- Field analysis data
- Field observations on sampling event
- Name of sampler
- Weather conditions

## **9.0 Quality Control and Assurance**

- 9.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the project-specific SAP. The goal of the QA program should be to ensure precision, accuracy, representativeness, completeness, and comparability in the project sampling program.
- 9.2 Quality control (QC) requirements for sample collection are dependent on project-specific sampling objectives. The project-specific SAP will provide requirements for sample preservation and holding times, container types, sample packaging and shipment, as well as requirements for the collection of various QC samples such as trip blanks, field blanks, equipment rinse blanks, and field duplicate samples.

## **10.0 Data and records management**

- 10.1 Records will be maintained in accordance with SOP 3-03, Recordkeeping, Sample Labelling, and Chain-of-Custody. Various forms are required to ensure that adequate documentation is made of the sample collection activities. These forms may include:
- Sample Collection Records;
  - Field logbook;
  - Chain-of-custody forms; and
  - Shipping labels.

- 10.2 Sample collection records (Attachment 1) will provide descriptive information for the purging process and the samples collected at each monitoring well.
- 10.3 The field logbook is kept as a general log of activities and should not be used in place of the sample collection record.
- 10.4 Chain-of-custody forms are transmitted with the samples to the laboratory for sample tracking purposes.
- 10.5 Shipping labels are required is sample coolers are to be transported to a laboratory by a third party (courier service).

## 11.0 Attachments or References

### Attachment 1 – Groundwater Sampling Collection Record

ASTM Standard D5088. 2008. *Standard Practice for Decontamination of Field Equipment Used at Waste Sites*. ASTM International, West Conshohocken, PA. 2008. DOI: 10.1520/D5088-02R08. [www.astm.org](http://www.astm.org).

Environmental Protection Agency, United States (EPA). 1982. *Handbook for Sampling and Sample Preservation of Water and Wastewater*. EPA-600/4-82-029. Cincinnati: EPA Office of Research and Development, Environmental Monitoring and Support Laboratory.

EPA. 1992. *RCRA Groundwater Monitoring Draft Technical Guidance*. EPA/530/R-93/001. Office of Solid Waste. November.

EPA. 1996. *Ground Water Issue: Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures*. EPA/540/S-95/504. Office of Solid Waste and Emergency Response. April.

EPA. 1997. *Test Methods for Evaluating Solid Waste, Physical/Chemical Method (SW-846)*. 3rd ed., Final Update IIIA. Office of Solid Waste. Online updates at: <http://www.epa.gov/epaoswer/hazwaste/test/new-meth.htm>.

NAVSEA T0300-AZ-PRO-010. *Navy Environmental Compliance Sampling and Field Testing Procedures Manual*. August 2009.

SOP 3-03, *Recordkeeping, Sample Labelling, and Chain-of-Custody*.

SOP 3-05, *IDW Management*.

SOP 3-06, *Equipment Decontamination*.

<i>Author</i>	<i>Reviewer</i>	<i>Revisions (Technical or Editorial)</i>
Mark Kromis Program Chemist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)

## Attachment 1

### Groundwater Sample Collection Record



Well ID: \_\_\_\_\_

### Groundwater Sample Collection Record

Client: _____	Date: _____	Time: Start _____ am/pm
Project No: _____		Finish _____ am/pm
Site Location: _____		
Weather Conds: _____	Collector(s): _____	

#### 1. WATER LEVEL DATA: (measured from Top of Casing)

a. Total Well Length \_\_\_\_\_ c. Length of Water Column \_\_\_\_\_ (a-b) Casing Diameter/Material \_\_\_\_\_

b. Water Table Depth \_\_\_\_\_ d. Calculated Well Volume (see bag-4) \_\_\_\_\_

#### 2. WELL PURGEABLE DATA

a. Purge Method: \_\_\_\_\_

b. Acceptance Criteria defined (see SAP or Work Plan)

- Minimum Required Purge Volume (@ \_\_\_\_\_ well volumes) \_\_\_\_\_
- Maximum Allowable Turbidity \_\_\_\_\_ NTUs
- Stabilization of parameters \_\_\_\_\_ %

c. Field Testing Equipment used: Make \_\_\_\_\_ Model \_\_\_\_\_ Serial Number \_\_\_\_\_

Time (min)	Volume Removed (gal)	Temp. (°C)	pH s.u.	Spec. Cond. (µS/cm)	DO (mg/L)	ORP (mv)	Turbidity (NTU)	Flow Rate (ml/min)	Drawdown (m)	Color/Odor/etc.

d. Acceptance criteria pass/fail Yes No N/A (continues on back)

Has required volume been removed ☐ ☐ ☐

Has required turbidity been reached ☐ ☐ ☐

Have parameters stabilized ☐ ☐ ☐

If no or N/A - Explain below.

#### 3. SAMPLE COLLECTION:

Method: \_\_\_\_\_

Sample ID	Container Type	No. of Containers	Preservation	Analysis Req.	Time

Comments \_\_\_\_\_

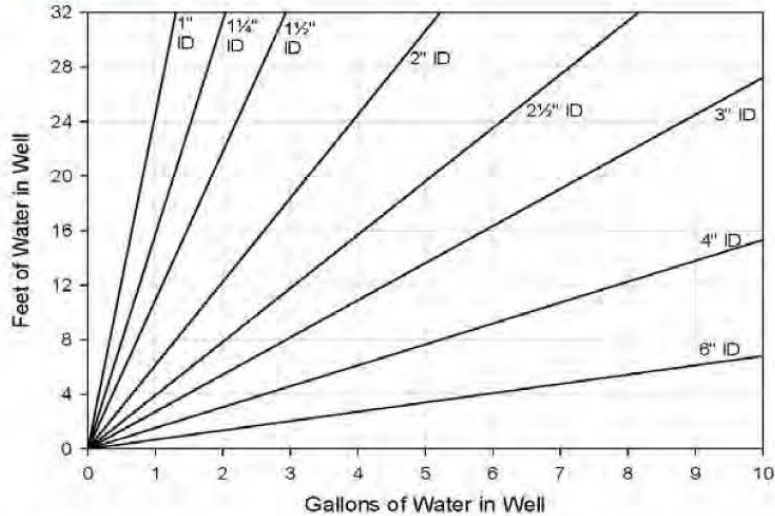
Signature \_\_\_\_\_

Date \_\_\_\_\_



### Purge Volume Computation

Well ID:



Volume / Linear Ft. of Pipe		
ID (in)	Gallon	Liter
¼	0.0025	0.0097
⅜	0.0057	0.0217
½	0.0102	0.0386
¾	0.0229	0.0869
1	0.0408	0.1544
1¼	0.0637	0.2413
1½	0.0918	0.3475
2	0.1632	0.6178
2½	0.2550	0.9653
3	0.3672	1.3900
4	0.6528	2.4711
6	1.4688	5.5600

(continued from front)

[illegible]

Signature \_\_\_\_\_ Date \_\_\_\_\_

# Surface and Subsurface Soil Sampling Procedures

## Procedure 3-21

### 1.0 Purpose and Scope

- 1.1 This standard operating procedure (SOP) describes the procedures for soil sampling. The procedure includes surface and subsurface sampling by various methods using hand auguring, test pit, direct-push, and split-spoon equipment.
- 1.2 The procedure includes soil sampling for volatile organic compounds (VOCs). For project specific information (e.g. sampling depths, equipment to be used, and frequency of sampling), refer to the Sampling and Analysis Plan (SAP), which takes precedence over these procedures. Surface soil sampling, typically considered to be up to two feet below ground surface by EPA standards, is typically accomplished using hand tools such as shovels or hand augers. Test pit samples are considered subsurface samples, although normally collected via hand tools similar to surface soil sampling or by excavation machinery. Direct-push and split-spoon sampling offer the benefit of collecting soil samples from a discrete or isolated subsurface interval, without the need of extracting excess material above the target depth. These methods dramatically reduce time and cost associated with disposal of material from soil cuttings when compared to test pit sampling. In addition, direct-push and split-spoon sampling methods can obtain samples at targeted intervals greater than 15 feet in depth, allowing for discrete depth soil sampling while speeding up the sampling process. Direct-push methods work best in medium to fine-grained cohesive materials such as medium to fine sands, silts, and silty clay soils. Split-spoon sampling works well in all types of soil, but is somewhat slower than direct-push methods. Samples are composited so that each sample contains a homogenized representative portion of the sample interval. Due to potential loss of analytes, samples for volatile analysis are not composited. Samples for chemical analysis can be collected by any of the above-mentioned sampling methods, as disturbed soil samples. Undisturbed samples are collected, sealed, and sent directly to the laboratory for analysis. For undisturbed samples, the samples are not homogenized.

### 2.0 Safety

- 2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the project Health and Safety Plan (HASP). In the absence of a HASP, work will be conducted according to the Contract Task Order (CTO) Work Plan (WP) and/or direction from the **Site Safety Officer (SSO)**.
- 2.2 Before soil sampling commences, appropriate entities (e.g. DigSafe, local public works departments, company facilities) must be contacted to assure the anticipated soil sampling locations are marked for utilities, including electrical, telecommunications, water, sewer, and gas.

### 3.0 Terms and Definitions

None.

### 4.0 Interferences

- 4.1 Low recovery of soil from sampling equipment will prevent an adequate representation of the soil profile and sufficient amount of soil sample. If low recovery is a problem, the hole may be offset and re-advanced, terminated, or continued using a larger diameter sampler.



- 4.2 Asphalt in soil samples can cause false positive results for hydrocarbons. To ensure samples are free of asphalt, do not collect samples that may contain asphalt. If the collection of samples potentially containing asphalt is unavoidable, note the sampling depths at which the presence of asphalt are suspected.
- 4.3 Instrumentation interferences addressed in SOPs for Calibration of the Photoionization Detector (PID), Headspace Screening for Total Volatile Organics, and Equipment Decontamination must also be considered.
- 4.4 Cross contamination from sampling equipment must be prevented by using sampling equipment constructed of stainless steel that is adequately decontaminated between samples.

## **5.0 Training and Qualifications**

### **5.1 Qualifications and Training**

The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

### **5.2 Responsibilities**

- 5.2.1 The CTO Manager is responsible for ensuring that soil sampling activities comply with this procedure. The CTO Manager is responsible for ensuring that all personnel involved in soil sampling shall have the appropriate education, experience, and training to perform their assigned tasks.
- 5.2.2 The Program Quality Manager is responsible for ensuring overall compliance with this procedure.
- 5.2.3 The Field Manager is responsible for ensuring that all soil sampling activities are conducted according to this procedure.
- 5.2.4 All Field Personnel are responsible for the implementation of this procedure.

## **6.0 Equipment and Supplies**

The depth at which samples will be collected and the anticipated method of sample collection (direct-push, split-spoon, hand auger, shovel, or test pits) will be presented in the SAP. The following details equipment typically needed for soil sampling, based on the various methods. See the SAP for specific detail of equipment and supply needs.

- 6.1 Depending on the nature of suspected contamination, field screening instrumentation may be used for direct sampling. Appropriate instrumentation and calibration standards should be available. If volatile organic contaminants are suspected and a PID will be used, refer to the equipment and instrumentation listed in SOP 3-20 Operation and Calibration of a Photoionization Detector. Equipment in this SOP includes but is not limited to:
  - PID/FID;
  - Calibration gas; and
  - Tedlar® gas bags (for calibration).
- 6.2 If field screening methods include jar headspace screening for volatile organics, refer to the equipment and procedure in SOP 3-19 Headspace Screening for Total VOCs. Equipment in this SOP includes but is not limited to:
  - Clean soil ("drillers jars") jars; and
  - Aluminium foil.

- 6.3 Appropriate decontamination procedures must be followed for sampling equipment. Refer to SOP 3-06 Equipment Decontamination. Equipment in this SOP includes but is not limited to:
- Phosphate-free detergent;
  - Isopropyl Alcohol;
  - Tap water;
  - Deionized Ultra-Filtered (DIUF) Water;
  - Plastic buckets or washbasins;
  - Brushes; and
  - Polyethylene sheeting.
- 6.4 The following general equipment is needed for all soil sampling, regardless of method:
- Stainless steel bowls;
  - Stainless steel trowels;
  - Appropriate sample containers for laboratory analysis;
  - Personal Protective Equipment (PPE);
  - Logbook;
  - Cooler and ice for preservation; and
  - Stakes and flagging to document sampling location.
- 6.5 The following additional equipment is needed for volatile organic sampling:
- Electronic pan scale and weights for calibration; and
  - Syringes or other discrete soil core samplers.
- 6.6 The following additional equipment may be needed for surface and test pit soil sampling:
- Hand Auger
- 6.7 The following additional equipment may be needed for soil sampling from direct push and/or split-spoon equipment:
- Tape measure or folding carpenter's rule for recording the length of soil recovered.

Note: All subsurface drilling equipment will be provided and maintained by the subcontractor.

## **7.0 Procedure**

### **7.1 General Soil Sampling Procedure for All Soil Sampling Methods**

- 7.1.1 Record the weather conditions and other relevant on-site conditions.
- 7.1.2 Select the soil sampling location, clear vegetation if necessary, and record the sampling location identification number and pertinent location details.
- 7.1.3 Verify that the sampling equipment is properly decontaminated, in working order, and situated at the intended sampling location.

- 7.1.4 Place polyethylene sheeting on the ground and assemble all necessary sampling equipment on top of it. Cover surfaces onto which soils or sampling equipment will be placed (i.e. tables with polyethylene sheeting).
- 7.1.5 Follow the appropriate procedures listed below for either surface, split-spoon, direct push, or test pit sample collection (7.2, 7.3, 7.4, and 7.5 respectively).
- 7.1.6 Collect soil samples according to procedures listed in Section 7.6 depending on project specific analyses.
- 7.1.7 Record date/time, sample ID, and sample descriptions in the field logbook or field form. A sketch or description of the location may also be recorded so the sample location can be re-constructed, especially if the location will not be recorded using global positioning satellite (GPS) equipment.
- 7.1.8 Immediately label the sample containers and place them on ice, if required for preservation. Complete the chain-of-custody form(s) as soon as possible.
- 7.1.9 Dispose of all excess excavated soil in accordance with the SAP.
- 7.1.10 If required, mark the sample location with a clearly labelled wooden stake or pin flag. If the location is on a paved surface, the location may be marked with spray paint.
- 7.1.11 Decontaminate the sampling equipment according to SOP 3-06 Equipment Decontamination.

## **7.2 Surface Sampling**

- 7.2.1 The criteria used for selecting surface soil locations for sampling may include the following:
  - Visual observations (soil staining, fill materials);
  - Other relevant soil characteristics;
  - Site features;
  - Screening results;
  - Predetermined sampling approach (i.e. grid or random); and
  - Sampling objectives as provided in the SAP.
- 7.2.2 The following procedures are to be used to collect surface soil samples. Surface soils are considered to be soils that are up to two feet below ground surface, though state regulations and project objectives may define surface soils differently; therefore, the SAP should be consulted for direction on the depth from which to collect the surface soil samples. Sampling and other pertinent data and information will be recorded in the field logbook and/or on field forms. Photographs may be taken as needed or as specified in the SAP.
  1. Gently scrape any vegetative covering until soil is exposed. Completely remove any pavement.
  2. Remove soil from the exposed sampling area with a trowel, hand auger, or shovel. Put soils within the sampling interval in a stainless steel bowl for homogenizing. Monitor the breathing zone and sampling area as required in the HASP.
  3. For VOC analyses, collect representative soil samples directly from the recently-exposed soil using a syringe or other soil coring device (e.g., TerraCore®, EnCore®). Follow procedures in Section 7.6.1 for VOC sampling.
  4. Collect sufficient soil to fill all remaining sample jars into a stainless steel bowl. Homogenize the soil samples to obtain a uniform soil composition which is representative of the total soil sample collected according to the following procedure:
    - a) Remove all rocks and non-soil objects using a stainless steel spoon or scoop.

- b) Form a cone shaped mound with the sample material, then flatten the cone and split the sample into quarters.
- c) Use the stainless steel spoon/scoop to mix the quarter samples that are opposite.
- d) After mixing the opposite quarters, reform the cone shaped mound.
- e) Repeat this procedure a minimum of five (5) times, removing any non-soil objects and breaking apart any clumps.

### 7.3 Split-Spoon Sampling

- 7.3.1 At each boring location, the frequency and depth of split-spoon samples will be determined from the SAP. Split-spoon samples may be collected continuously, intermittently, or from predetermined depths.
- 7.3.2 Split-spoon samplers shall be driven into undisturbed soil by driving the spoon ahead of the drill augers/casing. In cohesive soils, or soils where the borehole remains open (does not collapse), two split-spoon samples may be taken prior to advancing the augers/casing.
- 7.3.3 After split-spoons are retrieved, open the split-spoon and measure the recovery of soil. If a PID will be used for screening, immediately scan the recovered sample for VOCs using the PID. Scan the recovered soil boring by making a hole in the soil with a decontaminated trowel and placing the PID inlet very close to the hole. Be very careful not to get soil on the tip of the PID. Take PID readings every 6 inches along the split-spoon and/or in any areas of stained or disturbed soil. Record the highest PID reading and the depth at which it was observed along with all other pertinent observations. If required in the SAP, VOC and headspace samples should be collected (see Section 7.6.1) prior to logging the sample.
- 7.3.4 If headspace screening for VOCs is required in the SAP, collect a soil sample (as defined in the SAP) and perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.
- 7.3.5 Soils collected using the split-spoon sampler will be logged by the field representative using the procedure required in the SAP.
- 7.3.6 Collect the remainder of the sample volume required into a stainless steel bowl. Homogenize the soil so the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.
- 7.3.7 The SAP may specify that intervals to be sent to the laboratory be determined by visual observation and/or highest PID screening or headspace results, which can only be determined once the boring is complete. In this instance, a VOC sample should be collected at each interval. The remainder of the soil from that interval will be set aside in a clearly labelled stainless steel bowl covered with aluminium foil. Once the boring has been completed and the sample interval has been determined, the remainder of the soil can be homogenized according to Section 7.2 and submitted for laboratory analysis.
- 7.3.8 Once a boring is complete and all required samples have been collected, the boring must be completed as specified in the SAP (e.g., completed as a monitoring well, backfilled with bentonite, etc).

### 7.4 Direct Push Sampling

At each boring location, the frequency of direct-push samples will be determined from the SAP. Typically, samples with direct-push equipment are collected in 4 foot (ft) intervals, but smaller (e.g., 2 ft) and larger (e.g., 5 ft) intervals are also possible.

- 1. Sample using Macro-Core samplers with acetate liners to obtain discrete soil samples at the depths specified in the SAP.
- 2. Cut open the acetate liner. If required in the SAP, immediately scan the recovered soil boring for VOCs using a PID by making a hole in the soil with a decontaminated trowel and placing the PID inlet very close to the hole. Be very careful not to get soil on the tip of the PID. Take PID readings every 6 inches along the split-spoon and/or in any areas of stained or disturbed soil. Record the

highest PID reading and the depth at which it was observed along with all other pertinent observations. VOC and headspace samples, if required in the SAP should be collected (see Section 7.6.1) prior to logging the sample.

3. If required in the SAP, collect a soil sample (as defined in the SAP) and perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.
4. Soils collected using the direct-push sampler will be logged by the by the field representative using the procedure required in the SAP.
5. Collect the remainder of the sample into a stainless steel bowl. Homogenize the soil collected so that the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.
6. Once a boring is complete and all required samples have been collected, the boring must be completed as specified in the SAP (e.g., completed as a monitoring well, backfilled with bentonite, etc).

## **7.5 Test Pit Sampling**

7.5.1 Excavate the test pit to the desired depth.

7.5.2 Using the excavator bucket, collect soil samples as specified in the SAP. Collect a sample and perform screening analyses as required by the SAP. If VOCs contamination is suspected, perform headspace screening according to SOP 3-19 Headspace Screening for Total VOCs.

7.5.3 Collect the sample from center of the bucket to avoid potential contamination from the bucket.

7.5.4 VOC samples should also be collected from an undisturbed section soil in the excavator bucket. The top layer of exposed soil should be scraped away just prior to collecting the VOC samples.

7.5.5 Collect the remainder of the sample volume required into a stainless steel bowl. Homogenize the soil so the material is uniform in composition and representative of the total soil sample collected. Follow homogenizing techniques as described in Section 7.2.

7.5.6 Dispose of all excavated soil according to the SAP.

## **7.6 Sample Collection Methods**

### **7.6.1 Volatile Organics Sampling**

For soils collected for analyses of volatile organics, including Volatile Petroleum Hydrocarbons (VPH) or other purgable compounds, a closed system is maintained. From collection through analysis, the sample bottles are not opened. The bottle kit for a routine field sample for these analyses will typically include three 40-mL VOA vials and one soil jar. Two 40-mL VOA vials will contain either 5 mL reagent water or 5 mL sodium bisulfate and magnetic stir bars (i.e., low level vials). The third VOA vial will contain 15 mL methanol with no magnetic stir bar (i.e., high level vial). These vials are usually provided by the laboratory and are pre-weighed, with the tare weight recorded on the affixed sample label. No additional sample labels are affixed to the VOA vials, as addition of a label would alter the vial weight. All information is recorded directly on the sample label using an indelible marker. The soil jar is provided for percent solids determination. For VOC or VPH analyses, samples are collected prior to sample homogenization. Collect the VOC sample in accordance with the procedure described below.

1. Determine the soil volume necessary for the required sample weight, typically 5 grams:
  - a) Prepare a 5 mL sampling corer (e.g., Terra Core®) or cut-off plastic syringe.
  - b) Tare the sampler by placing it on the scale, and zeroing the scale.
  - c) Draw back the plunger to the 5 gram mark or 5mL (5cc) mark on cut-off syringe, and insert the open end of the sampler into an undisturbed area of soil with a twisting motion, filling the

sampler with soil. Note the location of the plunger with respect to the milliliter (cc) or other graduation printed on the sampler.

- d) Weigh the filled sampler, and remove or add soil until the desired weight is obtained. Note the location of the plunger which corresponds to this weight. Do not use this sample for laboratory analysis.
2. Once the required soil volume has been determined, pull the plunger back to this mark and hold it there while filling the syringe for each sample.
3. Collect 5 grams of soil using the cut-off syringe or Terra Core® sample device. Extrude the 5-grams of soil into one of the low level 40-mL VOA vials. Quickly wipe any soil from the threads of the VOA vial with a clean Kimwipe® and immediately close the vial. It is imperative that the threads be free from soil or other debris prior to replacing the cap on the vial in order to maintain the closed system necessary for the analysis.
4. Gently swirl the vial so that all of the soil is fully wetted with the preservative.
5. Fill the other low level 40 mL VOA vial in this manner.
6. Repeat the process for the high level VOA vials, only for the high level VOA vial three 5 gram aliquots (i.e., 15 grams total) should be extruded into the high level VOA vial.

NOTE: Depending on the laboratory, some high level VOA vials only contain 5 mL or 10 mL of methanol. If this is the case, either 5 grams total or 10 grams total, respectively, should be extruded into the high level VOA vial. In other words, the mass of soil in grams should be identical to the volume of methanol in mL (i.e., 1:1 ratio of soil to methanol).

7. Collect any additional QC sample collected (e.g., field duplicate, MS, and MSD) in the same manner as above.
8. Fill the 4-oz glass jar with soil from the same area for percent moisture determination.

#### 7.6.2 Soil Sampling Method (All other analyses except VOC/VPH)

When all the required soil for a sampling location has been obtained, the soil can be homogenized as described in section 7.2. Collect sufficient volume to fill all of the remaining sample containers at least  $\frac{3}{4}$  full for all other analyses. Homogenize the soil in a decontaminated stainless steel bowl, removing rocks, sticks, or other non-soil objects and breaking apart any lumps of soil prior to filling the remaining sample containers.

NOTE: Soil samples must contain greater than 30% solids for the data to be considered valid.

## 8.0 Quality Control and Assurance

- 8.1 Sampling personnel should follow specific quality assurance guidelines as outlined in the SAP. Proper quality assurance requirements should be provided which will allow for collection of representative samples from representative sampling points. Quality assurance requirements outlined in the SAP typically suggest the collection of a sufficient quantity of field duplicate, field blank, and other samples.
- 8.2 Quality control requirements are dependent on project-specific sampling objectives. The SAP will provide requirements for equipment decontamination (frequency and materials), sample preservation and holding times, sample container types, sample packaging and shipment, as well as requirements for the collection of various quality assurance samples such as trip blanks, field blanks, equipment blanks, and field duplicate samples.



## 9.0 Records, Data Analysis, Calculations

All data and information (e.g., sample collection method used) must be documented on field data sheets, boring logs, or within site logbooks with permanent ink. Data recorded may include the following:

- Weather conditions;
- Arrival and departure time of persons on site;
- Instrument type, lamp (PID), make, model and serial number;
- Calibration gas used;
- Date, time and results of instrument calibration and calibration checks;
- Sampling date and time;
- Sampling location;
- Samples collected;
- Sampling depth and soil type;
- Deviations from the procedure as written; and
- Readings obtained.

## 10.0 Attachments or References

SOP 3-06, *Equipment Decontamination*

SOP 3-19, *Headspace Screening for Total VOCs*

SOP 3-20, *Operation and Calibration of a Photoionization Detector*

Author	Reviewer	Revisions (Technical or Editorial)
Robert Shoemaker Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)

# Sediment Sampling

## Procedure 3-22

### 1.0 Purpose and Scope

1.1 Sediment contamination is a widespread environmental problem that can pose a threat to a variety of aquatic ecosystems. Sediment functions as a reservoir for common contaminants such as pesticides, herbicides, polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), and metals such as lead, mercury, and arsenic. Contaminated sediments represent a hazard to aquatic life through direct toxicity, as well as to aquatic life, wildlife, and human health through bioaccumulation. Accurate assessment of environmental hazards posed by sediment contamination depends in large part on the accuracy and representativeness of sediment collection and analyses (U.S. EPA, 2001).

1.2 Selection and proper use of sediment sampling equipment is essential to the collection of accurate, representative sediment data that will meet the project Data Quality Objectives (DQOs). Most sediment collection devices are designed to isolate and consistently retrieve a specified volume and surface area of sediment, from a required depth below the sediment surface, with minimal disruption of the integrity of the sample and no contamination of the sample. Maintaining the integrity of the collected sediment, for the purposes of the measurements intended, is a primary concern in most studies because disruption of the sediment's structure could change its physiochemical and biological characteristics, thereby influencing the bioavailability of contaminants and the potential toxicity of the sediment (U.S. EPA, 2001).

When selecting the type of sediment sampling equipment to be used for an event, the project DQOs as well as the sediment characteristics should be considered. Related to the project DQOs is the desired depth of sediment sampling. For monitoring and assessment studies where historical contamination is not the focus, the upper 10 to 15 centimeters (cm) is typically the horizon of interest, as this is the horizon that generally contains the most recently deposited sediments and most epifaunal and infaunal organisms (U.S. EPA, 2001). The 0-6 inches interval for sediments with less than two feet of water is also used for human health risk assessment purposes. Sampling of these horizons can usually be done with grab samplers. However, if sediment contamination is being related to organism exposures (e.g. benthic macroinvertebrates and/or fish), or if characterization of deeper sediments is important for comparison of recent surficial versus historical contamination, then more precise sampling of sediment depths might be needed, and a hand corer may be more suitable (U.S. EPA, 2001).

1.3 This standard operating procedure (SOP) describes the procedure for the collection of sediment samples using the Petite Ponar<sup>®</sup> Grab Sampler, Ekman Bottom Grab Sampler, and Wildco<sup>®</sup> Hand Corer (or similar sampling devices). The applicability of each of the sediment samplers is described below.

The Petite Ponar<sup>®</sup> Grab Sampler is used to collect sediment samples in:

- Firm, hard bottoms such as sand, gravel, consolidated marl, and clay
- Mixtures of sand, stones, and coarse debris
- Soft or mucky sediments

The Ekman Bottom Grab Sampler is used to collect sediment samples in:

- Soft, finely divided littoral bottoms free from vegetation and intermixtures of sand, stones, and other coarse debris
- Bottoms composed of finely divided mulch, mud, muck, or submerged fine peaty materials

The Wildco® Hand Corer is used:

- To collect sediment samples for geological characterizations and dating
- To collect sediment samples for programs where it is important to maintain an oxygen-free environment for the sample during collection
- To collect sediment samples from a deeper depth than a grab sampler, and to characterize the depth of contamination at a site
- To investigate the historical input of contaminants to aquatic systems
- To collect sediment samples in semi-consolidated and soft sediment

Pictures and exploded diagrams of the Petite Ponar Grab Sampler, Ekman Bottom Grab Sampler, and Wildco® Hand Corer are presented in Figures 1, 2, and 3, respectively.

- 1.4 This procedure is the Program-approved professional guidance for work performed by Resolution Consultants under the Comprehensive Long-Term Environmental Action Navy (CLEAN) contract (Contract Number N62470-11-D-8013).
- 1.5 As guidance for specific activities, this procedure does not obviate the need for professional judgment. Deviations from this procedure while planning or executing planned activities must be approved in accordance with Program requirements for technical planning and review.

## **2.0 Safety**

- 2.1 Depending upon the site-specific contaminants, various protective programs must be implemented prior to sampling the first location. All **field sampling personnel** responsible for sampling activities must review the project-specific health and safety plan (HASP) paying particular attention to the control measures planned for the sampling tasks. Conduct preliminary area monitoring of sample locations to determine the potential hazard to field sampling personnel. If significant contamination is observed, minimize contact with potential contaminants in both the vapor phase and solid or liquid matrix through the use of respirators and disposable clothing.
- 2.2 Observe standard health and safety practices according to the project-specific HASP. Suggested minimum protection during sediment sampling activities includes inner disposable vinyl gloves, outer chemical-protective nitrile gloves, and waders (if applicable). Refer to the project-specific HASP for the required PPE.
- 2.3 Handle all sediments removed from potentially contaminated locations in accordance with the IDW handling procedures in SOP 3-05, IDW Management.
- 2.4 Depending upon the type of contaminant expected or determined in previous sampling efforts, employ the following safe work practices:
- If sampling from a boat, all sampling personnel should wear personal flotation devices (PFDs) when in the boat, and should follow all health and safety protocols for working in a boat presented in the project-specific HASP.
  - Lifting the samplers into the boat, dumping its contents, and washing those contents may require leaning over the side of the boat. Care should be taken to keep the boat in proper balance at all times during sampling.
  - Severe injury to fingers or hands can be caused by movement of the lever arms of the Petite Ponar® Grab Sampler. Do not handle or move the Petite Ponar® Grab Sampler unless the safety pin is fully inserted in the locking holes.
  - Severe injury to fingers or hands can be caused by the closing of the sharpened scoops of the Ekman Bottom Grab Sampler. Handle the Ekman Bottom Grab Sampler very carefully when the springs are set and the cable loops are hooked (armed) on the Twin-Pin™ pins on the release mechanism. Do not “arm” the Ekman Bottom Grab Sampler until the sampler is ready

to be used. The Ekman Bottom Grab Sampler spring-loaded jaws are potentially dangerous; extreme care must be exercised when setting the jaws. To prevent injury (and to extend the life of the springs), unhook both springs from their scoop buttons after each sampling session.

### **3.0 Terms and Definitions**

None.

### **4.0 Training and Qualifications**

- 4.1 The **Contract Task Order (CTO) Manager** is responsible for ensuring that sediment sampling activities comply with this procedure. The **CTO Manager** is responsible for ensuring that all field sampling personnel involved in sediment sampling shall have the appropriate education, experience, and training to perform their assigned tasks.
- 4.2 The **Program Quality Manager** is responsible for ensuring overall compliance with this procedure.
- 4.3 The **Field Manager** is responsible for ensuring that all field sampling personnel follow these procedures.
- 4.4 **Field sampling personnel** are responsible for the implementation of this procedure.
- 4.5 The field sampler and/or task manager is responsible for directly supervising the sediment sampling procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data collected during sampling.

### **5.0 Equipment and Supplies**

- 5.1 For sediment sampling using all types of equipment, the following supplies are required:
- Stainless steel bowls
  - Stainless steel hand trowels, spoons, spatulas, and scoops
  - Munsell Color Chart
  - Particle size chart
- 5.2 Petite Ponar<sup>®</sup> Grab Sampler
- 3/16" braided polyester line
  - Auxiliary weights
- 5.3 Ekman Bottom Grab Sampler
- 11 oz split messenger
  - 3/16" braided polyester line
  - Extension Handle
  - Auxiliary weights
- 5.4 Wildco<sup>®</sup> Hand Corer
- 3/16" braided polyester line
  - Extension handle
  - Stainless steel core catchers (for normal sediments)
  - Eggshell<sup>™</sup> core catchers (for wet sediments)
  - Stainless steel nose piece
  - Cellulose acetate butyrate (CAB) liners
  - Core liner end caps
  - Core liner cutter
  - Geologists table

- Auxiliary weights

## 6.0 Procedure

6.1 Depending on the characteristics of the site being investigated, sediment samples may be collected from a boat, or by sampling personnel in waders. In all instances, sediment sampling should begin from the most downstream location and proceed to the most upstream location. If sediment samples are collocated with surface water samples, the surface water sample should be collected prior to the sediment sample in order to avoid increased turbidity from displaced sediment. Regardless of the type of sediment sampling equipment used, documentation of field observations and collection activities should be recorded on the sediment sampling sheet or electronic data collection device. The following observations should be recorded on the sediment sampling form (see Attachment 1) for all sediment sampling activities:

- Sample location
- Weather conditions and other relevant site conditions
- Depth of water to the nearest 0.1 foot. A surveyor rod may be used. If the surveyor rod is used, minimize water turbulence and do not disturb any sediment.
- Physical characteristics of the water body such as estimated current speed (stagnant, slow, medium, or fast) and direction, odor, color, presence of any dead vegetation, surface sheens, etc.
- Sediment color according to the Munsell Color Chart
- Sediment grain size according to a particle size chart

Specific procedures for the collection of sediment samples using the Petite Ponar<sup>®</sup> Grab Sampler, Ekman Bottom Grab Sampler, and Wildco<sup>®</sup> Hand Corer are presented below.

### 6.2 Petite Ponar<sup>®</sup> Grab Sampler

- 6.2.1 Inspect the sampler to ensure all parts are in good working condition.
- 6.2.2 Decontaminate the sampler according to the procedures in SOP 3-06, Equipment Decontamination.
- 6.2.3 Attach the 3/16" braided polyester line to the sampler by looping the line through the clevis at the top center of the lever arms and tying securely. Tie the other end of the line to the boat (if applicable), or make sure to hold on to the other end of the line. Strong, tight knots (e.g. bowline, two half hitches) are essential for operator safety and to prevent losing the sampler. If necessary, attach the auxiliary weights to the sampler according to the manufacturer's directions.
- 6.2.4 Insert the Pinch-Pin<sup>™</sup> into its hole in the lever arms, making sure to firmly push the Pinch-Pin<sup>™</sup> into the hole. As long as the line is taught, the Pinch-Pin<sup>™</sup> will stay in its place. When the line becomes the least bit slack (e.g. when the sampler hits the bottom), the Pinch-Pin<sup>™</sup> spring will force the Pinch-Pin<sup>™</sup> out of its hole, allowing the scoops to close.
- 6.2.5 Just before lowering the grab into the water, and with the line taught, remove the safety pin so the closing mechanism will release when the sampler is on the bottom. Make sure to keep the line taught, as any loss of tension in the line will cause the Pinch-Pin<sup>™</sup> to pop out, closing the sampler.
- 6.2.6 Lower the sampler into the water in a slow and controlled fashion, especially during the final 1-2', such that the bow wave is minimized, thus minimizing the dispersal of fine material on the sediment surface. At no time should the sampler be allowed to "free fall" down through the water column.

- 6.2.7 Once the sampler has reached the bottom, release the tension on the line, and allow the sampler to sink into the sediment momentarily. The release of tension on the line will cause the Pinch-Pin™ to pop out.
- 6.2.8 Collect the sample by pulling on the line, which will cause the lever arms to drive the scoops into the sediment in a closing motion. Keep pulling on the line in a controlled fashion until the scoops drive through the sediment and close.
- 6.2.9 Once the sampler scoops have closed, continue pulling on the line in a controlled fashion in order to retrieve the sampler back to the surface. When the sampler reaches the surface, lift it clear and bring it above a decontaminated stainless steel bowl. Inspect the sampler to ensure that an acceptable sample has been collected (See Figure 4). If the sample is not acceptable, discard the sample in an area that is not proximal or upstream to the area or subsequent areas that are being sampled.
- 6.2.10 Prior to sampling and sample homogenization, the overlying water in the sampler should be siphoned off, and not decanted (U.S. EPA 2001).
- 6.2.11 If acid volatile sulfide/simultaneously extracted metals (AVS/SEM) samples are to be collected, open the top screens of the sampler and collect the AVS/SEM sample directly from the sediment contained in the sampler according to the procedures specified in the project-specific SAP.
- 6.2.12 If volatile organic compound (VOC) samples are to be collected, open the top screens of the sampler and collect the VOC samples by inserting a syringe, Terra Core sampler, or other VOC sampling device directly into the undisturbed sediment contained within the sampler, making sure to follow all VOC sampling procedures specified in the project-specific SAP. Once the VOC samples have been collected, collect an additional aliquot for the VOC percent solids sample directly from the undisturbed sediment contained within the sampler.
- 6.2.13 Once the AVS/SEM and VOC samples have been collected (or if AVS/SEM and VOC samples are not required), open the sampler by pulling the two scoops open, taking care to keep hands and fingers away from the sharpened edges of the scoops, and allow the sediment to exit the sampler into the decontaminated stainless steel bowl.
- 6.2.14 If additional aliquots are necessary to provide adequate sample volume, repeat steps 6.2.3 through 6.2.12 until an adequate sample volume has been collected, taking care to deploy the sampler to an area that is proximal and upstream, but not on top of, the previous sample location.
- 6.2.15 Once an adequate sample volume has been collected, homogenize the sample in the stainless steel bowl, record the sediment sample information on the Sediment Sample Collection Form (see Attachment 1), and collect the sediment samples according to the procedures specified in the project-specific SAP (typically in order of decreasing volatility).
- 6.3 Ekman Bottom Grab Sampler with the 11 oz Split Messenger
  - 6.3.1 Inspect the sampler to ensure all parts are in good working condition.
  - 6.3.2 Decontaminate the sampler according to the procedures in SOP 3-06, Equipment Decontamination.
  - 6.3.3 Attach the 3/16" braided polyester line to the sampler by passing the line through the trip mechanism and knotting it securely below the underlying plate. Thread the 11 oz split messenger on the line, and tie the other end of the line to the boat (if applicable), or make sure to hold on to the other end of the line. Strong, tight knots (e.g. bowline, two half hitches) are essential to prevent losing the sampler. If necessary, attach the auxiliary weights to the sampler according to the manufacturer's directions.



- 6.3.4 Set the spring on the side of the sampler by hooking the end of the spring onto one scoop button and stretching the spring to reach the second scoop button. Repeat this procedure with the spring on the other side of the sampler.
- 6.3.5 Arm the scoops by hooking one cable loop to one Twin-Pin™ pin in the trip assembly on the top of the sampler. The white ball on the cable can be used as a hand grip to assist getting the cable loop hooked onto the Twin-Pin™ pin. Repeat for the opposite cable loop. The sampler is now armed and dangerous. Do not allow anything to come in contact with the trip assembly at the top of the sampler, as this may cause a sudden and unexpected closure of the sampler.
- 6.3.6 Lower the sampler into the water in a slow and controlled fashion, especially during the final 1-2', such that the bow wave is minimized, thus minimizing the dispersal of fine material on the sediment surface. At no time should the sampler be allowed to "free fall" down through the water column.
- 6.3.7 Once the sampler has reached the bottom, allow the sampler to settle momentarily. Once the sampler has settled, hold the line with just enough tension to keep it straight, and send the 11 oz split messenger down the line. Once the 11 oz split messenger impacts Twin-Pin™ strike pad in the trip assembly on the top of the sampler, the two cable loops will be released from the Twin-Pin™ pins, and the spring-loaded scoops of the sampler will automatically close.
- 6.3.8 Retrieve the sampler by pulling up the line in with a moderate, steady speed. When the sampler reaches the surface, lift it clear and bring it above a decontaminated stainless steel bowl. Inspect the sampler to ensure that an acceptable sample has been collected (See Figure 4). If the sample is not acceptable, discard the sample in an area that is not proximal or upstream to the area or subsequent areas that are being sampled.
- 6.3.9 Prior to sampling and sample homogenization, the overlying water in the sampler should be siphoned off, and not decanted (U.S. EPA 2001).
- 6.3.10 If AVS/SEM samples are to be collected, open the top lids of the sampler and collect the AVS/SEM sample directly from the sediment contained in the sampler according to the procedures specified in the project-specific SAP.
- 6.3.11 If VOC samples are to be collected, open the top lids of the sampler and collect the VOC samples by inserting a syringe, Terra Core sampler, or other VOC sampling device directly into the undisturbed sediment contained within the sampler, making sure to follow all VOC sampling procedures specified in the project-specific SAP. Once the VOC samples have been collected, collect an additional aliquot for the VOC percent solids sample directly from the undisturbed sediment contained within the sampler.
- 6.3.12 Once the AVS/SEM and VOC samples have been collected (or if AVS/SEM and VOC samples are not required), open the sampler by pulling on the white balls on both cables, opening the spring-loaded scoops and allowing the sediment to exit the sampler into the decontaminated stainless steel bowl. While the spring-loaded scoops are being held open, do not place hands or fingers inside or underneath the sampler.
- 6.3.13 If additional aliquots are necessary to provide adequate sample volume, repeat steps 6.3.4 through 6.3.11 until an adequate sample volume has been collected, taking care to deploy the sampler to an area that is proximal and upstream, but not on top of, the previous sample location.
- 6.3.14 Once an adequate sample volume has been collected, homogenize the sample in the stainless steel bowl, record the sediment sample information on the Sediment Sample Collection Form (see Attachment 1), and collect the sediment samples according to the procedures specified in the project-specific SAP (typically in order of decreasing volatility).

#### 6.4 Ekman Bottom Grab Sampler with the Extension Handle

- 6.4.1 Inspect the sampler to ensure all parts are in good working condition.
  - 6.4.2 Decontaminate the sampler according to the procedures in SOP 3-06, Equipment Decontamination.
  - 6.4.3 Attach the extension handle to the top of the sampler with machine bolts.
  - 6.4.4 Arm the sampler according to the procedures described in steps 6.3.3 and 6.3.4 above.
  - 6.4.5 Using the extension handle, lower the sampler to a point 4-6" above the sediment surface, and drop the sampler to the sediment, keeping the sampler vertical at all times.
  - 6.4.6 Trigger the trip assembly by depressing the button on the upper end of the extension handle. This will cause the two cable loops to be released from the Twin-Pin™ pins, and the spring-loaded scoops of the sampler will automatically close.
  - 6.4.7 While keeping the sampler vertical, bring the sampler over to a decontaminated stainless steel bowl. Inspect the sampler to ensure that an acceptable sample has been collected (See Figure 4). If the sample is not acceptable, discard the sample in an area that is not proximal or upstream to the area or subsequent areas that are being sampled.
  - 6.4.8 Collect samples according to the procedures described in steps 6.3.8 through 6.3.13 above.
- 6.5 Wildco® Hand Corer with the Push Handles
- 6.5.1 Inspect the sampler to ensure all parts are in good working condition:
    - Assemble and disassemble the core tube from the head and nose piece to make sure the threads are not binding. If the threads are binding, consult the manufacturer's directions.
    - Make sure that the CAB plastic liner can slide easily in and out of the core tube.
    - Make sure the bottom edge of the core tube and nose piece are sharp and free from nicks or dents. If necessary, file smooth using a round file.
    - Check the flutter valve for ease of movement.
    - Check the flutter valve seat to make sure it is clear of any obstruction, disfigurement, grease, and/or oil that could prevent a tight closure.
  - 6.5.2 Decontaminate the sampler according to the procedures in SOP 3-06, Equipment Decontamination.
  - 6.5.3 Screw the corer head onto the core tube, and screw the two handles onto the corer head.
  - 6.5.4 Insert a CAB plastic liner into the core tube, insert a core catcher onto the end of the CAB plastic liner (stainless steel for normal sediments, Eggshell™ for wet sediments), and screw the stainless steel nose piece onto the core tube. If using the hand corer from a boat, bridge, high dock, etc., be sure that the appropriate extension handle (5', 10' or 15') is attached to the corer head.
  - 6.5.5 Get in position over the sampling location. If wading in shallow water, be sure to approach the sample location from the downstream side. Line up the sampler, aiming it vertically for the point where the sample is being taken, and push the hand corer in a smooth continuous motion through the water and into the sediment. Increase the thrust as necessary to obtain the penetration desired. Do not hammer or pound the corer into the sediment.
  - 6.5.6 Retrieve the sample by pulling straight up on the handles, keeping the corer as vertical as possible. If the corer has not been completely submerged, close the flutter valve by hand and press it shut while the sample is being retrieved. The flutter valve must be kept very wet if it is to seal properly and prevent sample washout. If the substrate is gripping the corer too tightly, gently rock the top of the corer back and forth horizontally to increase the size of the hole created by the corer and reduce the pull-out suction.

- 6.5.7 Unscrew the nose piece from the corer and cap the bottom end of the CAB core liner. Release the flutter valve to free the CAB core liner, and slide the CAB core liner from the core tube. Cap the top of the CAB core liner and inspect the CAB core liner for recovery. If the recovery is adequate, proceed to step 6.5.8. If the recovery is not adequate, resample the location by repeating steps 6.5.3 through 6.5.7.
- 6.5.8 Bring the CAB core liner with the sediment sample over to the geologist table, keeping the core vertical. Place the CAB core liner on the geologist table and cut open with a core liner cutter. If AVS/SEM samples are to be collected, collect the AVS/SEM sample directly from the sediment contained in the core liner according to the procedures specified in the project-specific SAP. If VOC samples are to be collected, collect the VOC samples by inserting a syringe, Terra Core sampler, or other VOC sampling device directly into the sediment core. Consult the project-specific SAP for project-specific VOC sediment sampling procedures. Once the VOC samples have been collected, collect an additional aliquot for the VOC percent solids sample directly from the sediment core.
- 6.5.9 Once the AVS/SEM and VOC samples have been collected (or if AVS/SEM and VOC samples are not required), use a decontaminated stainless steel spoon to transfer the remaining sediment core into a decontaminated stainless steel bowl.
- 6.5.10 If additional aliquots are necessary to provide adequate sample volume, repeat steps 6.5.3 through 6.5.8 until an adequate sample volume has been collected, taking care to deploy the corer to an area that is proximal, but not on top of, the previous sample location.
- 6.5.11 Once an adequate sample volume has been collected, homogenize the sample in the stainless steel bowl, record the sediment sample information on the Sediment Sample Collection Form (see Attachment 1), and collect the sediment samples according to the procedures specified in the project-specific SAP (typically in order of decreasing volatility).
- 6.6 Wildco® Hand Corer with the Clevis and Line
  - 6.6.1 Inspect the corer as described in step 6.5.1 above.
  - 6.6.2 Decontaminate the sampler according to the procedures in SOP 3-06, Equipment Decontamination.
  - 6.6.3 Screw the corer head onto the core tube. Attach the 3/16" braided polyester line to the corer by passing the line through the clevis in the corer head and knotting it securely. Strong, tight knots are essential to prevent losing the corer. If necessary, attach the auxiliary weights to the sampler according to the manufacturer's directions.
  - 6.6.4 Insert a CAB plastic liner into the core tube, insert a core catcher onto the end of the CAB plastic liner (stainless steel for normal sediments, Eggshell™ for soupy sediments), and screw the stainless steel nose piece onto the core tube.
  - 6.6.5 Position the corer over the drop point and steady momentarily, making sure to keep the corer vertical at all times. Make sure to arrange the 3/16" braided polyester line to run freely. Since the corer's penetration is by simple gravity, it is important that there be no restraint on the corer during descent by stricture on the line. Keep a firm hold on the free end of the line, or tie it to the boat (if applicable) or some other permanent fixture.
  - 6.6.6 Drop the corer into the water, and allow the corer to free fall until it hits the sediment surface. The corer should not be dropped to depths greater than 20' to 30'. Dropping the corer to depths greater than 20' to 30' may result in the corer striking the sediment surface at an angle less than 90°, resulting in an unsatisfactory sample.
  - 6.6.7 Once the corer has entered the sediment and is no longer falling, draw the line taut, and then pull on the line to pull the corer from the sediment. Once the corer has been pulled free from

the sediment, bring the corer back to the surface by pulling up the line, using a smooth, hand-over-hand fashion. This movement automatically causes the flutter valve to close, preventing sample washout in all but the soupiest of sediments.

- 6.6.8 Once the corer has been returned to the surface, lift the corer clear of the water, being careful to keep the corer as vertical as possible at all times.
- 6.6.9 Collect the sediment sample according to the procedures outlined in steps 6.5.6 through 6.5.11 above.

## **7.0 Quality Control and Assurance**

- 7.1 Field personnel will follow specific quality assurance (QA) guidelines as outlined in the project-specific SAP. The goal of the QA program should be to ensure precision, accuracy, representativeness, completeness, and comparability in the project sampling program.
- 7.2 Quality control (QC) requirements for sample collection are dependent on project-specific sampling objectives. The project-specific SAP will provide requirements for sample preservation and holding times, container types, sample packaging and shipment, as well as requirements for the collection of various QC samples such as trip blanks, field blanks, equipment rinse blanks, and field duplicate samples.

## **8.0 Records, Data Analysis, Calculations**

- 8.1 Records will be maintained in accordance with SOP 3-03, Recordkeeping, Sample Labelling, and Chain-of-Custody. Various forms are required to ensure that adequate documentation is made of the sample collection activities. These forms may include:
- Sample Collection Records;
  - Field logbook;
  - Chain-of-custody forms; and
  - Shipping labels.
- 8.2 Sample collection records (Attachment 1) will provide descriptive information for the sediment samples collected at each location.
- 8.3 The field logbook is kept as a general log of activities and should not be used in place of the sample collection record.
- 8.4 Chain-of-custody forms are transmitted with the samples to the laboratory for sample tracking purposes.
- 8.5 Shipping labels are required is sample coolers are to be transported to a laboratory by a third party (courier service).

## **9.0 Attachments or References**

Attachment 1 – Sediment Sample Collection Record

Figure 1 – Petite Ponar® Grab Sampler and Exploded Diagram

Figure 2 – Ekman Bottom Grab Sampler (Large, Tall, and Standard Sizes) and Exploded Diagram

Figure 3 – Wildco® Hand Corer (with Case and Accessories) and Exploded Diagram

Figure 4 – Illustrations of Acceptable and Unacceptable Grab Samples

NAVSEA T0300-AZ-PRO-010. *Navy Environmental Compliance Sampling and Field Testing Procedures Manual*. August 2009.

U.S. Environmental Protection Agency (U.S. EPA). 2001. *Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual*. October.

Wildlife Supply Company. 2003. 2424- Hand Corer Instructions.

Wildlife Supply Company. 2004. Ekman Bottom Grabs Instructions and Maintenance.

Wildlife Supply Company. 2004. 1728-G30/ 1728-G40 Petite Ponar<sup>®</sup> Grab.

SOP 3-05, *IDW Management*.

SOP 3-06, *Equipment Decontamination*.

<i>Author</i>	<i>Reviewer</i>	<i>Revisions (Technical or Editorial)</i>
Robert Shoemaker Senior Scientist	Chris Barr Program Quality Manager	Rev 0 – Initial Issue (May 2012)

## Attachment 1

### Sediment Sample Collection Record

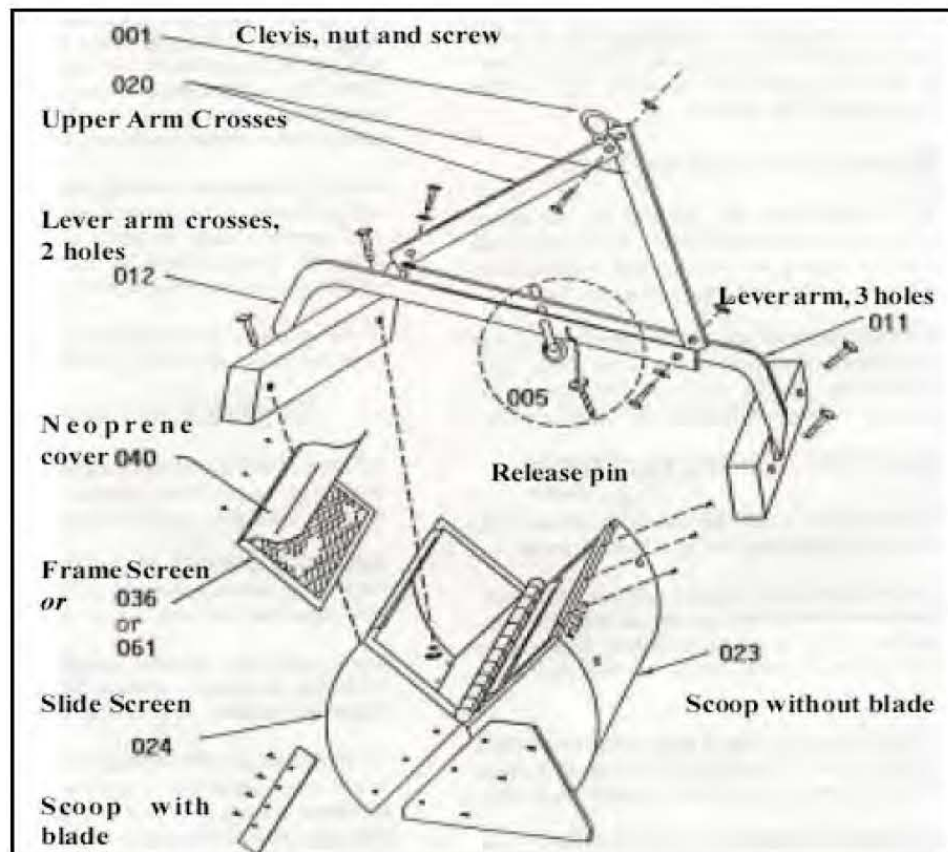


#### SEDIMENT SAMPLE COLLECTION FORM

Project Name:			
Date(s):			
Project #:		Date:	
Sample Location ID:		Time:	
Sample #:		Weather:	
Samplers:			
<b>Sample Information:</b>			
Sample Depth:		Sampling Device:	
Water Depth:			
Distance from River Bank:			
River Flow Rate:			
Field Decon:	Yes      No	Sample Type:	Grab      Composite
	Dedicated		
Munsell Color:			
Sample Description:			
Other physical characteristics of water body at sample location: (Water color, turbidity, odor, presence of sheens, dead/stressed vegetation)			
Sample Comments/Description:			

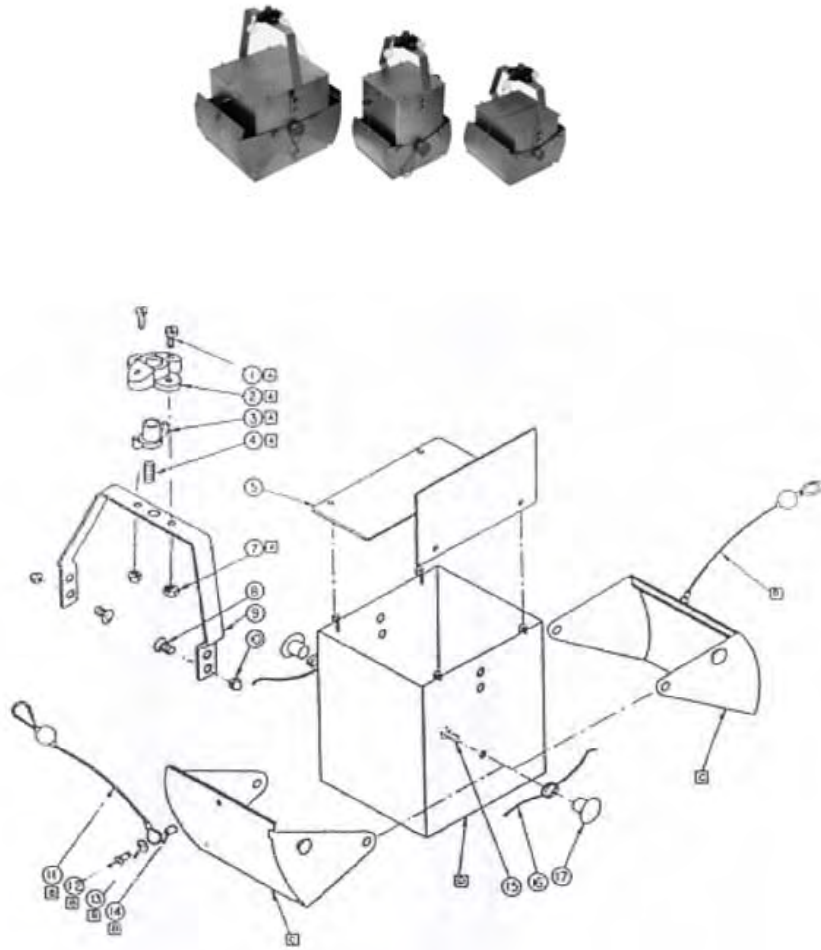


**Figure 1**  
**Petite Ponar® Grab Sampler and Exploded Diagram**



**Figure 2**

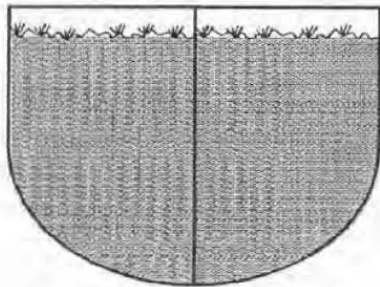
**Ekman Bottom Grab Sampler (Large, Tall, and Standard Sizes) and Exploded Diagram**



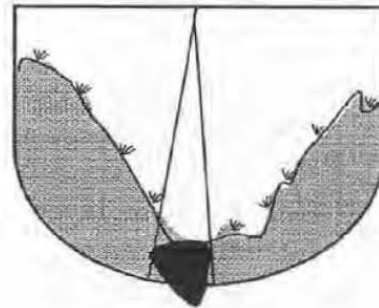
**Figure 3**  
**Wildco® Hand Corer (with Case and Accessories) and Exploded Diagram**



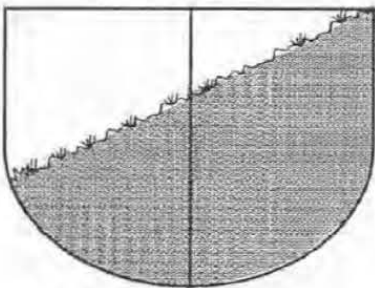
**Figure 4**  
**Illustrations of Acceptable and Unacceptable Grab Samples**



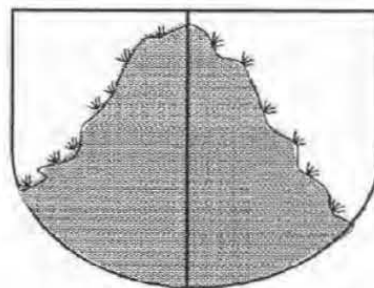
Acceptable if Minimum  
Penetration Requirement Met  
and Overlying Water is Present



Unacceptable  
(Washed, Rock Caught in Jaws)



Unacceptable (Canted with  
Partial Sample)



Unacceptable  
(Washed)

**Standard Operating Procedure SOP-3-24**  
**Water Quality Parameter Testing for Groundwater Sampling**

## **1.0 PURPOSE**

This standard operating procedure (SOP) represents minimum standard of practice. State and federal requirements may vary, and this SOP does not replace state and federal requirements that must be consulted before work begins. Further, if a project-specific work plan has been created, the work plan should be considered the ruling document. This SOP may be modified to meet specific regulatory, client, or project specific criteria.

If there are procedures whether it be from Resolution Consultants, state and/or federal that are not addressed in this SOP and are applicable to water quality parameter testing, then those procedures may be added as an appendix to the project-specific Sampling and Analysis Plan (SAP).

## **2.0 SCOPE**

This procedure provides guidance for expected sampling methods and protocols by all personnel related to the measurement of water quality parameters.

Field measurements of water quality parameters are commonly performed to evaluate surface water and groundwater. These tests are often performed to evaluate basic water quality parameters, to evaluate natural attenuation parameters, and to assess the presence of pore water entering a well.

As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved by either the Contract Task Order (CTO) Manager or the Quality Assurance (QA) Manager, and documented.

## **3.0 DEFINITIONS**

### **3.1 Barometric Pressure (BP)**

The density of the atmosphere, which varies according to altitude and weather conditions.

### **3.2 Conductivity/Specific Conductance**

A measure of the ability of water to pass electrical current, which increases with the amount of dissolved ionic substances (i.e., salts). Conductivity is inversely related to the resistance of a solution and is measured in units of mhos per centimeter (mhos/cm) (inverse ohms/cm, Siemens/cm). The conductivity of water increases with increasing temperature.



*Specific Conductance is corrected for 25 degrees Celsius (°C); for this reason, it is best to record Specific Conductance. If Conductivity is recorded, the temperature of the sample MUST recorded.*

### **3.3 Dissolved Oxygen (DO)**

The amount of oxygen present in water and available for respiration. DO is typically measured in milligrams per liter (mg/L). Oxygen is less soluble in warm and salty waters, so the instrument compensates the apparent percent saturation for changes in temperature and conductivity. Most probes measure the current resulting from the electrochemical reduction of oxygen (at a gold cathode) diffusing through a selective membrane. Because oxygen is being removed from the sample to perform the measurement, sample flow is required to prevent false low readings due to depletion of oxygen in the solution in front of the probe. Optical DO probes do not remove oxygen from the sample and are less affected by salts. The common range of DO in groundwater is 0.0 to 3.0 mg/L. Measurements outside of this range suggest that the meter may not be operating correctly.

### **3.4 Nephelometric Turbidity Unit (NTU)**

The measurement of light passing through a sample based on the scattering of light caused by suspended particles.

### **3.5 pH**

A measure of acidity and alkalinity of a solution using a logarithmic scale on which a value of 7 represents neutrality, lower numbers indicate increasing acidity, and higher numbers are increasingly basic.

### **3.6 Oxidation-Reduction Potential (ORP)**

Also known as redox or eH, ORP is a measurement of the potential for a reaction to occur, which generally indicates the oxygen status of a sample. The probe consists of a platinum electrode, the potential of which is measured with respect to a reference electrode that rapidly equilibrates with the potential of the sample solution. A positive value indicates that oxygen is present. A negative value indicates an anaerobic environment or reducing condition. For this reason, negative ORP readings should be associated with DO readings of less than 0.5 mg/l; with negative ORP readings the water may exhibit a sulfur odor or gray color. Positive ORP readings should be associated with DO readings greater than 0.5 mg/L and lack of sulfur odors. Because of the complex relationship between ORP and temperature, no compensation is attempted; it is thus best to report both the ORP and temperature of a water sample.

### **3.7 Total Dissolved Solids**

A measure of the quantity of materials in water that are either dissolved or too small to be filtered.

### **3.8 Turbidity**

Measure of the clarity of water in NTUs. Potable water typically has NTU values between 0.0 and 0.3 NTUs, depending on the state or regulatory program.

## **4.0 RESPONSIBILITIES**

The CTO Manager, or designee, is responsible for ensuring that these standard groundwater sampling activities are followed and shall review all groundwater sampling forms at the conclusion of a sampling event. The CTO Manager is responsible for ensuring that all personnel involved in monitoring well sampling shall have the appropriate education, experience, and training to perform their assigned tasks. The QA Manager or Technical Director is responsible for ensuring overall compliance with this procedure. The Field Manager is responsible for ensuring that all project field staff follows these procedures.

Field sampling personnel are responsible for the implementation of this procedure. Personnel are required to be knowledgeable of the procedures in this SOP. Training and familiarization with this SOP shall be documented in the training file for each employee. The field sampler and/or Field Manager is responsible for directly supervising the calibration procedures to ensure that they are conducted according to this procedure, and for recording all pertinent data. If deviations from the procedure are required because of anomalous field conditions, they must first be approved by the CTO Manager, QA Manager, or Technical Director and then documented in the field logbook and associated report or equivalent document.

## **5.0 PROCEDURES**

### **5.1 Purpose**

The procedures will vary depending on parameters being measured, method of sampling, and the method of measurement used. The information here is a general guidance and the site-specific documents and manufacturer manuals supersede these procedures.

### **5.2 Cautions**

Improper use of water quality testing equipment could result in equipment damage or compromised sampling results. Personnel should be trained to operate the test equipment being used for a field operation and should be trained in the proper techniques for collecting and

logging water quality parameters. Personnel should also be able to recognize problems with test equipment and have someone available for basic troubleshooting and repair.

### **5.3 Interferences**

During field testing, water quality data that is documented from field testing equipment may be influenced by certain outside factors that are unrelated to the actual site water quality. Such parameters and equipment include the following:

#### **pH Meters**

- Coatings of oils, greases, and particles may impair the electrode's response. Pat the electrode bulb dry with lint-free paper or cloth and rinse with de-ionized water. For cleaning hard-to-remove films, use isopropyl alcohol very sparingly so that the electronic surface is not damaged.
- Poorly buffered solutions with low specific conductance (less than 200 microsiemens per centimeter) may cause fluctuations in the pH readings. Equilibrate electrode by immersing in several aliquots of sample before taking pH.

#### **Dissolved Oxygen**

- Dissolved gases (e.g., hydrogen sulfide, halogens, sulfur dioxide) are a factor with the performance of DO probes. The effect is less pronounced on optical DO meters. Meter type and potential interferences should be considered based on potential sulfate/sulfide or nitrate/nitrite reducing environments.
- Exposure of the sample to the atmosphere will cause elevated DO measurements.

#### **Turbidity Meter**

- If the weather is warm and humidity is high, condensation may collect on the cuvet. To avoid this, allow the sample to warm and dry the outside of the cuvet before making the measurement. One method used to accomplish this is to place the cuvet against one's body (armpits work well).

#### **Temperature**

- Sample temperature will change rapidly when there are significant differences between the sample and ambient air.

## 5.4 Apparatus and Materials

Field personnel shall consult the site work plan and SAP to review the equipment requirements for the sampling procedures to be followed during the sampling effort. The specific apparatus and materials required will depend on the water quality parameters being monitored. Table 1 shows the common equipment used in water quality parameter testing.

**Table 1**  
**Water Quality Parameter Testing — Common Equipment**

Water Quality Parameter Instrument	Calibration Standards Required	Other Equipment
pH Meter	Yes - 2 or 3 Point Standards depending on groundwater range. Calibration must cover the range to be measured. If samples are above or below typical buffer standards (4, 7 and 10), special order buffers that fall outside groundwater pH range.	Container or flow thru cell for holding sample
Specific Conductance	Yes	Container or flow thru cell for holding sample
ORP Meter	Yes	Container or flow thru cell for holding sample
Turbidity Meter	Yes	Container or flow thru cell for holding sample
DO	No	Container or flow thru cell for holding sample
Thermometer	No	Container or flow thru cell for holding sample
Flow Rate	No	Calibrated Container

**Notes:**

ORP = Oxidation-Reduction Potential  
 DO = Dissolved Oxygen

## 5.5 Instrument or Method Calibration

Most monitoring instruments require calibration before use, and this calibration must be conducted in the field under the ambient climatic conditions that will be present during field sampling. Calibration of monitoring instruments shall be performed in accordance with the manufacturer's specifications and recorded in the provided form in Attachment 1. Site-specific instrument calibration requirements should be specified in the SAP. The following minimum calibration requirements apply to the various types of meters used to gather water quality measurements.

**Initial Calibration (IC):** Before use, the instrument or meter electronics are adjusted (manually or automatically) to a theoretical value (e.g., DO saturation) or a known value of a

calibration standard. An IC is performed in preparation for the first use of an instrument or if a calibration verification does not meet acceptance criteria.

**Initial Calibration Verification (ICV):** The instrument or meter calibration is checked or verified directly following IC by measuring a calibration standard of known value as if it were a sample and comparing the measured result to the calibration acceptance criteria for the instrument/parameter. If an ICV fails to meet acceptance criteria, immediately recalibrate the instrument using the applicable initial calibration procedure or remove it from service.

**Continuing Calibration Verification (CCV):** After use, the instrument or meter calibration is checked or verified by measuring a calibration standard of known value as if it were a sample and comparing the measured result to the calibration acceptance criteria for the instrument/parameter.

### 5.5.1 Calibration Checks

Calibration checks are conducted by measuring a known standard. They must be completed after calibration and should be performed at least one other time (i.e., after lunch) and anytime suspect measurements are encountered. Table 2 provides general acceptance ranges to be used during calibration checks. If a meter is found to be outside of the acceptance range, the meter **must** be recalibrated. If the meter remains out of range, the project manager and/or the supplier of the meter should be contacted to determine alternative measures.

**Table 2**  
**Calibration Check Acceptance Limits**

Parameter	Acceptance Criteria
Dissolved Oxygen	±0.3 mg/L of the theoretical oxygen solubility
Oxidation-Reduction Potential	±10 mv from the theoretical standard value at that temperature
pH	±0.2 Standard pH Units
Specific Conductance	±5% of the standard
Turbidity	0.1 to 10 NTU: ±10% of the standard 11 to 40 NTU: ±8% of the standard 41 to 100 NTU: ±6.5% of the standard

**Notes:**

mg/L = milligrams per liter  
 mv = millivolts  
 NTU = nephelometric turbidity units

### 5.5.2 Possible and Suspected Ranges

The concentration for each parameter range should be known so that concentrations outside of the range can be noted. Table 3 presents the maximum range of the parameter in groundwater. The table also presents the suspected range. Measurements outside of the maximum/minimum range should be considered in error and the measurement method should be checked. Concentrations outside the normal range should be treated as suspect but may be the result of contaminant impact. For example, a pH of 2.0 would be out of the normally suspected range for groundwater but not at a site impacted with an acid.

**Table 3**  
**Minimum and Maximum Result Ranges**

Parameter	Units	Possible Min	Possible Max	Normal Min	Normal Max	Notes
						The colder the sample, the higher the DO reading.
Dissolved Oxygen	mg/L	0.0	14.6 (0°C) 10.1 (15°C) 8.3 (2°C)	0.0	5	DO greater than 1 mg/L, ORP positive should not have sulfur odor, sulfide, ferrous iron and/or gray color.  DO less than 1 mg/L, ORP negative, may have sulfur odor, sulfide, ferrous iron and/or gray color.
pH	SU	0	14	5	9	pH values exceeding 10 could indicate grout contamination
ORP	mv					DO greater than 1 mg/L, ORP positive should not have sulfur odor, sulfide, ferrous iron and/or gray color.  DO less than 1 mg/L, ORP negative, may have sulfur odor, sulfide, ferrous iron and/or gray color.
Specific Conductance	µS/cm			varies	varies	
Temperature	°C	0	100	5	30	
Turbidity	NTU	0	Greater than 1,000	0	Greater than 1,000	50 NTU or greater suggests cloudiness.

**Notes:**

mg/L = milligrams per liter  
 °C = degrees Celsius  
 DO = dissolved oxygen  
 SU = standard units  
 ORP = oxidation reduction potential  
 mv = millivolts  
 mS/cm = micro Siemens per cm  
 NTU = nephelometric turbidity units



### 5.5.3 Field Instruments and Calibration Criteria

The calibration acceptance criteria for each instrument are summarized in Table 4 along with special considerations related to each field instrument.

**Table 4**  
**Calibration Check Acceptance Limits**

Parameter	Acceptance Criteria
Dissolved Oxygen	±0.3 mg/L of the theoretical oxygen solubility.
Oxidation-Reduction Potential	±10 mv from the theoretical standard value at that temperature.
pH	±0.2 Standard pH Units
Specific Conductance	±5% of the standard
Turbidity	0.1 to 10 NTU: ±10% of the standard
	11 to 40 NTU: ±8% of the standard
	41 to 100 NTU: ±6.5% of the standard

**Notes:**

mg/L = milligrams per liter  
 mv = millivolts  
 NTU = nephelometric turbidity units

#### pH Meters

- For the most accurate of pH measurements, pH meters should receive a three-point calibration. However, if a two-point calibration will bracket the groundwater pH of the site, a two-point calibration is acceptable. Three-point calibrations typically include calibrating to solutions of pH 7.00, 4.00, and 10.00. If groundwater pH is outside the calibration range of the solution standards, special buffers must be ordered to bracket the pH. Some meters will report the slope of the calibration and this may be used in checking the meter calibration (refer to the meter's manual). When performing an ICV, the result must be within +/- 0.2 pH units of the stated buffer value.
- pH meters should be calibrated across the range of values to be measured. The maximum and minimum calibration solutions shall be outside the range of anticipated values. For example, if the expected range is between 7.50 and 9.00, the 7.00 and the 10.00 standard should be used for calibration. Perform the IC using at least two buffers, and always use the pH 7.00 buffer first. A reading that is above the maximum (or below the minimum) calibration standard is an estimate only and is not valid. This condition requires obtaining a new standard that is above (or below) the reported value, depending on the measurement.

- A percent slope of less than 90 percent indicates a bad electrode that must be changed or repaired. If percent slope cannot be determined, or the manufacturer's optimum specifications are different, follow the manufacturer's recommendation for maintaining optimum meter performance.

### **Specific Conductivity Meters**

- For IC, when the sample measurements are expected to be 100 microsiemens per centimeter ( $\mu\text{S}/\text{cm}$ ) or greater, use two standard potassium chloride (KCl) solutions that bracket the range of expected sample conductivities. Calibrate the instrument with the first standard. Verify the calibration of the instrument with the second standard, bracketing the range of expected sample values.
- If the instrument can be calibrated with more than one standard, choose additional calibration standards within the range of expected sample values.
- When the sample measurements are expected to be less than 100  $\mu\text{S}/\text{cm}$ , a lower bracket is not required, but one standard (KCl) solution that is within the range of expected measurements must be used for the IC and the ICV.
- Accept the calibration if the meter reads within +/- 5 percent of the value of any calibration standard used to verify the calibration.
- Most field instruments read conductivity directly. Record all readings and calculations in the calibration records.
- For CCV, check the meter with at least one KCl standard with a specific conductance in the range of conductivity measured in environmental samples. The reading for the calibration verification must also be within +/- 5 percent of the standard value.
- If new environmental samples are encountered outside the range of the IC, verify the instrument calibration with two standards bracketing the range of sample values. If these calibration verifications fail, recalibrate the instrument.

### **Dissolved Oxygen Meters**

- Before calibrating, check the probe membrane for bubbles, tears, or wrinkles. These conditions require replacement of the membrane in accordance with the manufacturer's directions.
- If the meter provides readings that are off-scale, will not calibrate, or drift, check the leads, contacts, etc., for corrosion and/or short circuits. These conditions require replacement maintenance in accordance with the manufacturer's directions.
- Most DO meters must be calibrated based on an environment of 100 percent humidity and a known elevation and barometric pressure (BP).
- For 100 percent humidity, place the probe in the calibration container with a moist towel and allow the probe to remain, undisturbed, for 10 to 20 minutes.
- The IC is an air calibration at 100% saturation. Before use, verify the meter calibration in water-saturated air to make sure it is properly calibrated and operating correctly. Make a similar verification at the end of the day or sampling event. Follow the manufacturer's instructions for your specific instrument. Allow an appropriate warm up period before IC. Wet the inside of the calibration chamber with water, pour out the excess water (leave a few drops), wipe any droplets off the membrane/sensor and insert the sensor into the chamber (this ensures 100 percent humidity). Allow adequate time for the DO sensor and the air inside the calibration chamber to equilibrate. Once the probe/calibration chamber is stable at ambient temperature, check the air temperature and determine, from the DO versus temperature table (see Attachment 2) what DO should measure. The acceptance criterion for DO ICV is +/- 0.3 mg/L.
- Use the same procedure as above for CCV.

### **ORP Meters**

- Verify electrode response before use in the field.
- Equilibrate the standard solution to the temperature of the sample. The standard solution is based on a 25°C temperature; however, the calibration solution standard's value will require adjustment based on the temperature.

- Immerse the electrodes and gently stir the standard solution in a beaker (or flow cell). Turn the meter on, placing the function switch in the millivolt (mv) mode.
- Let the electrode equilibrate and record the reading to the nearest millivolt. The reading must be within  $\pm 10$  mv from the theoretical redox standard value at that temperature. If not, determine the problem and correct it before proceeding. Switch to temperature display and read the value.
- Record the mv reading and temperature in the field notebook or in form. Rinse the electrode with distilled water and proceed with the sample measurement, unless using a flow cell. If a flow cell is used, rinse between sample locations.

### **Turbidity Meters**

- Perform an initial calibration using at least two primary standards.
- If the instrument cannot be calibrated with two standards, calibrate the instrument with one standard and verify with a second standard.
- Perform an ICV by reading at least one primary standard as a sample. The acceptance criterion for the ICV depends on the range of turbidity of the standard value:
  1. Standard Value = 0.1 to 10 NTU: the response must be within 10 percent of the standard;
  2. Standard Value = 11 to 40 NTU: the response must be within 8 percent of the standard;
  3. Standard Value = 41 to 100 NTU: the response must be within 6.5 percent of the standard; and
  4. Standard Value greater than 100 NTU: the response must be within 5 percent of the standard.
- Determining the Values of Secondary Standards: Use only those certified by the manufacturer for a specific instrument. Secondary standards may be used for CCVs.

To initially determine the value of a secondary standard, assign the value that is determined immediately after an ICV or verification with primary standards. This is done by reading the secondary standard as a sample. This result must be within the manufacturer's stated tolerance range and  $\pm 10$  percent of the assigned standard value. If the  $\pm 10$  percent criterion is not met, assign this reading as the value of the standard. If the reading is outside the manufacturer's stated tolerance range, discard the secondary standard.

- CCV: Perform a CCV using at least one primary or secondary standard. The calibration acceptance criteria are the same as those for an ICV.

## **5.6 Direct Measurements**

Direct measurements with meters are the most common methods and can be accomplished by placing a sample in a container with the probe or by allowing the water to flow past the probe in a flow cell. The use of a flow-through cell improves measurement quality by allowing the constant flow of water over the probes and reduces interaction of the sample with the atmosphere. Sample cups should be avoided. The quantity of samples, timing, and methodology should be described in the project SAP.

Following calibration of required probes, connect the bottom flow-cell port to the discharge line of the pump. Connect the top port to a discharge line directed to a bucket to collect the purge water. Allow the flow cell to completely fill. As the water flows over the probe, record the measurements. Continue to record the measurements at regular intervals, as specified in the SAP.

When the ambient air temperatures are much higher or lower than the temperature of the water sample, it is best to keep the length of tubing between the wellhead and the flow cell as short as possible to prevent heating or cooling of the water. Tubing and flow-through cell should not be exposed to direct sunlight, particularly in the summer, if at all possible, to avoid heating of water samples.

## **5.7 Data Acquisitions, Calculations, and Data Reduction**

### **5.7.1 Specific Conductivity Correction Factors**

If the meter does not automatically correct for temperature (i.e., read Specific Conductivity) record Conductivity and adjust for temperature upon returning to the office. The following equation can be used to convert Conductivity to Specific Conductivity.

$$K = \frac{(Km)(C)}{1 + 0.0191(T - 25)}$$

Where:

- K = Conductivity in  $\mu\text{mhos/cm}$  at  $25^{\circ}\text{C}$   
 Km = Measured conductivity in  $\mu\text{mhos/cm}$  at T degrees Celsius  
 C = Cell constant  
 T = Measured temperature of the sample in degrees Celsius;

If the cell constant is 1, the formula for determining conductivity becomes:

$$K = \frac{(Km)}{1 + 0.0191(T - 25)}$$

### 5.7.2 Percentage Difference Calculation

For evaluating slope of readings from either a flow cell or a sample cup.

$$\%Difference = \frac{(Highest\ Value - Lowest\ Value)}{(Highest\ Value)} \times 100$$

### 5.7.3 Convert mm mercury (mmHG) to inches mercury (inHG)

$$mmHG = inHG \times 25.4$$

### 5.7.4 True Barometric Pressure

For converting BP obtained from a public domain source that is expressed in BP at sea level to BP at the subject site.

$$TrueBP = (BP) - \frac{(2.5 \times [Local\ Altitude])}{100}$$

Where: BP is in mmHG and Local Altitude is in feet

Example: BP at site A is 30.49 inHg and elevation is 544 feet, calculate TrueBP



Convert inHG to mmHg:

$$\text{mmHg} = 30.49 \text{ inHg} \times 25.4 = 774.4 \text{ mmHg}$$

Calculate True BP:

$$\text{TrueBP} = (774.4 \text{ mmHg}) - [2.5 * (544 / 100)] = 774.4 - 13.6 = 760.8 \text{ mmHg}$$

## **6.0 RECORDS**

Data will be recorded promptly, legibly, and in indelible ink on the appropriate logbooks and forms. At the completion of a field effort, all logbooks, field data forms, and calibration logs shall be scanned and made electronically available to the project team. The original field forms, calibrations logs, and log book will be maintained in the project file.

## **7.0 HEALTH AND SAFETY**

Detailed Health and Safety requirements can be found in the site specific Health and Safety Plan. Ensure that a Safe Work Assessment and Permit form is filled out daily prior to any work in the field and reviewed with all project personnel in a daily safety brief.

Safety glasses with side shields or goggles and disposable gloves shall be worn during calibration activities.

## **8.0 REFERENCES**

None

## **9.0 ATTACHMENTS**

Attachment 1: Example Field Instrument Calibration Form

Attachment 2: Solubility of Oxygen at Given Temperatures

Attachment 3: Example Field Data Form

**Attachment 1**  
**Example Field Instrument Calibration Form**

# Field Instrument Calibration Form

Calibrated by: \_\_\_\_\_  
Date: \_\_\_\_\_

Equipment (Make/Model/Serial#): \_\_\_\_\_  
Equipment (Make/Model/Serial#): \_\_\_\_\_

pH (su) Standard: $\pm 0.2$ standard units				DO (mg/L) Standard: $\pm 0.3$ mg/L of theoretical*			
Initial Calibration		Initial Calibration Verification		IC (Temp: )		ICV (Temp: )	
Hach SL	Reading	Pine SL	Reading	Saturation (%)	Reading (%)	Theoretical (mg/L)	Reading (mg/L)
pH7				100			
pH4							
Continuing Calibration Verification				CCV (Temp: )			
			Acceptable Variance (Y/N)	Saturation (%)	Reading (%)	Deviation	Acceptable Variance (Y/N)
pH7				100			
pH4				Theoretical (mg/L)	Reading (mg/L)	Deviation	Acceptable Variance (Y/N)
ORP (mV) Standard: NA				Turbidity (ntu) Standard: $\pm 10\%$ of Standard			
IC (Zobell SL: )		ICV (Pine SL: )		Initial Calibration			
TCS (Std/Temp)	Reading	TCS (Std/Temp)	Reading	Standard		Reading	
CCV (Zobell SL: )				Continuing Calibration Verification			
TCS (Std/Temp)	Reading	Deviation	Acceptable Variance (Y/N)	Standard	Reading	Deviation	Acceptable Variance (Y/N)
Conductivity (ms <sup>c</sup> /cm) Standard: $\pm 5\%$ of standard value				Comments:			
IC (YSI SL: )		ICV (Pine SL: )					
Standard	Reading	Standard	Reading				
CCV (YSI SL: )							
Standard	Reading	Deviation	Acceptable Variance (Y/N)				

Notes: SL solution lot      TCS temperature corrected standard      Std standard      Temp temperature      su standard units      mV millivolts      % percent      mg/L milligrams per liter      ntu nephelometric turbidity units      °C degrees Celsius      ms<sup>c</sup>/cm millisiemens per centimeter (temperature corrected)      \* Theoretical value

**Attachment 2**  
**Solubility of Oxygen at Given Temperatures**

## Field Measurement of Dissolved Oxygen

Solubility of Oxygen in Water at Atmospheric Pressure			
Temperature	Oxygen Solubility	Temperature	Oxygen Solubility
°C	mg/L	°C	mg/L
0.0	14.621	26.0	8.113
1.0	14.216	27.0	7.968
2.0	13.829	28.0	7.827
3.0	13.460	29.0	7.691
4.0	13.107	30.0	7.559
5.0	12.770	31.0	7.430
6.0	12.447	32.0	7.305
7.0	12.139	33.0	7.183
8.0	11.843	34.0	7.065
9.0	11.559	35.0	6.950
10.0	11.288	36.0	6.837
11.0	11.027	37.0	6.727
12.0	10.777	38.0	6.620
13.0	10.537	39.0	6.515
14.0	10.306	40.0	6.412
15.0	10.084	41.0	6.312
16.0	9.870	42.0	6.213
17.0	9.665	43.0	6.116
18.0	9.467	44.0	6.021
19.0	9.276	45.0	5.927
20.0	9.092	46.0	5.835
21.0	8.915	47.0	5.744
22.0	8.743	48.0	5.654
23.0	8.578	49.0	5.565
24.0	8.418	50.0	5.477
25.0	8.263		

**Notes:**

The table provides three decimals to aid interpolation

Under equilibrium conditions, the partial pressure of oxygen in air-saturated water is equal to that of the oxygen in water saturated

°C = degrees Celsius

mg/L = milligrams per liter

**Attachment 3**  
**Example Field Data Form**



### WELL DEVELOPMENT & GROUNDWATER SAMPLING FORM

DATE:	JOB NUMBER:	EQUIPMENT (Make/Model #/Serial #):
PROJECT:	EVENT:	/ /
WELL ID:	LOCATION:	/ /
WEATHER CONDITIONS:	AMBIENT TEMP:	/ /
REVIEWED BY:	PERSONNEL:	/ /

WELL DIA:	<b>WELL DEVELOPMENT</b>
TOTAL DEPTH from TOC (ft.):	START: FINISH:
DEPTH TO WATER from TOC (ft.):	VOLUME PURGED (gal):
LENGTH OF WATER COL. (ft.):	<b>GROUNDWATER SAMPLING</b>
1 VOLUME OF WATER (gal):	START: FINISH:
3 VOLUMES OF WATER (gal):	VOLUME PURGED (gal):
	ANALYSIS:

<b>WELL DEVELOPMENT PARAMETERS</b>	<b>GW SAMPLING PARAMETERS</b>
Temperature: $\pm 1.0^{\circ}\text{C}$	Temperature: $\pm 0.2^{\circ}\text{C}$
pH: $\pm 0.5$ standard units	pH: $\pm 0.2$ standard units
Specific Conductance: $\pm 10\%$ of the past measurement	Specific Conductance: $\pm 5\%$ of the past measurement
Turbidity: relatively stable	DO: $\leq 20\%$ saturation
	ORP: $\pm 10$ millivolts
	Turbidity: $\leq 10$ NTU

#### IN-SITU TESTING

Circle one:	DEVELOPMENT	SAMPLING	<input type="checkbox"/> Bailer <input type="checkbox"/> Pump	Description:
Time (hh:mm):				
pH (units):				
Conductivity (mS/cm):				
Turbidity (NTU):				
DO (mg/L): YSI 556				
DO (mg/L): YSI 550				
Temperature (C°):				
ORP (mV):				
Volume Purged (gal):				
Depth to Water (ft):				
				Well Goes Dry While Purging <input type="checkbox"/>

#### SAMPLE DATA

	<input type="checkbox"/> Bailer <input type="checkbox"/> Pump	Description:
Sample ID	Date (m/d/y)	Time (hh:mm)
		Bottles (total to lab)
		Filtered (0.45 $\mu\text{m}$ )
		Remarks

Purging/Sampling Device Decon Process:

COMMENTS:

Purge water placed in drum#

Page \_ of \_

## **Appendix B**

### **Field Forms**

LOCATION OF BORING										JOB NO.		CLIENT		LOCATION					
										DRILLING METHOD:				BORING NO.					
														SHEET					
										SAMPLING METHOD:				OF					
										DRILLING									
										START		FINISH							
										TIME		TIME							
										DATE		DATE							
										CASING DEPTH									
DATUM										ELEVATION									
SAMPLER TYPE	INCHES DOWN INCHES RECOVERED	DEPTH OF CASING	SAMPLE NO. SAMPLE DEPTH	BLOWS/FT SAMPLER	WATER CONCENTRATIONS (PPM)	DEPTH IN FEET	SOIL GRAPH	SURFACE CONDITIONS											
						0													
						1													
						2													
						3													
						4													
						5													
						6													
						7													
						8													
						9													
						0													
						1													
						2													
						3													
						4													
						5													
						6													
						7													
						8													
						9													
						0													

# Field Instrument Calibration Form

Calibrated by: \_\_\_\_\_

Date: \_\_\_\_\_

Equipment (Make/Model/Serial#): \_\_\_\_\_

Equipment (Make/Model/Serial#): \_\_\_\_\_

pH (su) Standard: $\pm 0.2$ standard units				DO (mg/L) Standard: $\pm 0.3$ mg/L of theoretical*			
Initial Calibration		Initial Calibration Verification		IC (Temp: _____)		ICV (Temp: _____)	
Hach SL	Reading	Pine SL	Reading	Saturation (%)	Reading (%)	Theoretical (mg/L)	Reading (mg/L)
pH7				100			
pH4				CCV (Temp: _____)			
Continuing Calibration Verification				Saturation (%)	Reading (%)	Deviation	Acceptable Variance (Y/N)
Hach SL	Reading	Deviation	Acceptable Variance (Y/N)	100			
pH7				Theoretical (mg/L)	Reading (mg/L)	Deviation	Acceptable Variance (Y/N)
pH4							
ORP (mV) Standard: NA				Turbidity (ntu) Standard: $\pm 10\%$ of Standard			
IC (Zobell SL: _____)		ICV (Pine SL: _____)		Initial Calibration			
TCS (Std/Temp)	Reading	TCS (Std/Temp)	Reading	Standard		Reading	
CCV (Zobell SL: _____)				Continuing Calibration Verification			
TCS (Std/Temp)	Reading	Deviation	Acceptable Variance (Y/N)	Standard	Reading	Deviation	Acceptable Variance (Y/N)
Conductivity (ms <sup>c</sup> /cm) Standard: $\pm 5\%$ of standard value				Comments:			
IC (YSI SL: _____)		ICV (Pine SL: _____)					
Standard	Reading	Standard	Reading				
CCV (YSI SL: _____)							
Standard	Reading	Deviation	Acceptable Variance (Y/N)				

Notes: SL solution lot      su standard units      ntu Nephelometric Turbidity Units  
TCS temperature corrected standard      mV millivolts      °C degrees Celsius  
Std standard      % percent      ms<sup>c</sup>/cm millisiemens per centimeter (temperature corrected)  
Temp temperature      mg/L milligrams per liter      \* Theoretical value listed on Table FT 1500-1 (attached)



## WELL DEVELOPMENT & GROUNDWATER SAMPLING FORM

DATE:	JOB NUMBER:	EQUIPMENT (Make/Model #/Serial #):
PROJECT:	EVENT:	/ /
WELL ID:	LOCATION:	/ /
WEATHER CONDITIONS:	AMBIENT TEMP:	/ /
REVIEWED BY:	PERSONNEL:	/ /

WELL DIA:	WELL DEVELOPMENT
TOTAL DEPTH from TOC (ft.):	START: FINISH:
DEPTH TO WATER from TOC (ft.):	VOLUME PURGED (gal):
LENGTH OF WATER COL. (ft.):	GROUNDWATER SAMPLING
1 VOLUME OF WATER (gal):	START: FINISH:
3 VOLUMES OF WATER (gal):	VOLUME PURGED (gal):
	ANALYSIS:

WELL DEVELOPMENT PARAMETERS	GW SAMPLING PARAMETERS
Temperature: $\pm 1.0^{\circ}\text{C}$	Temperature: $\pm 0.2^{\circ}\text{C}$
pH: $\pm 0.5$ standard units	pH: $\pm 0.2$ standard units
Specific Conductance: $\pm 10\%$ of the past measurement	Specific Conductance: $\pm 5\%$ of the past measurement
Turbidity: relatively stable	DO: $\leq 20\%$ saturation
	ORP: $\pm 10$ millivolts
	Turbidity: $\leq 10$ NTU

### IN-SITU TESTING

Circle one: DEVELOPMENT SAMPLING	<input type="checkbox"/> Bailer <input type="checkbox"/> Pump	Description:
Time (hh:mm):		
pH (units):		
Conductivity (mS/cm):		
Turbidity (NTU):		
DO (mg/L): YSI 556		
DO (mg/L): YSI 550		
Temperature (C°):		
ORP (mV):		
Volume Purged (gal):		
Depth to Water (ft):		
		Well Goes Dry While Purging <input type="checkbox"/>

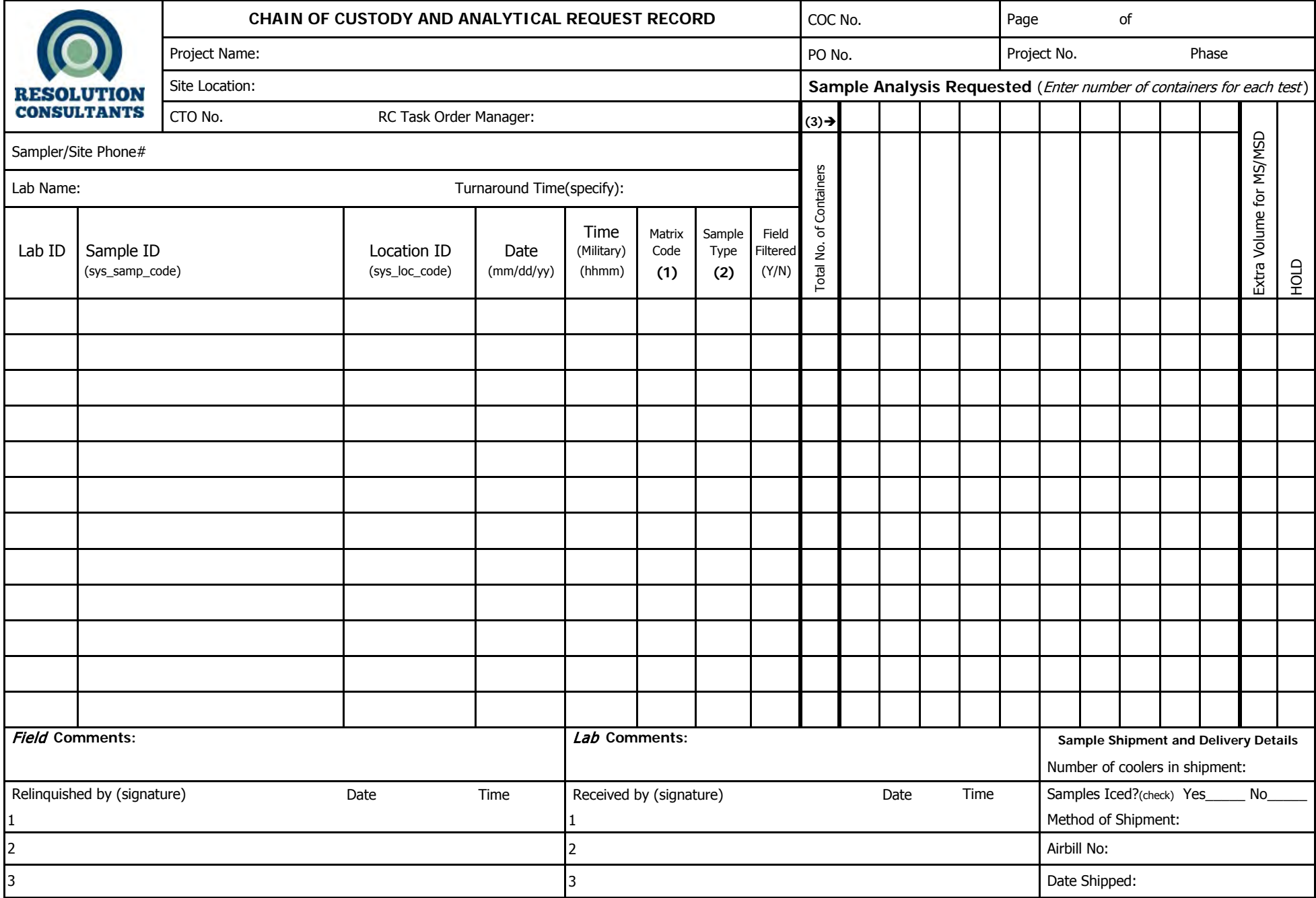
### SAMPLE DATA

	<input type="checkbox"/> Bailer <input type="checkbox"/> Pump	Description:			
Sample ID	Date (m/d/y)	Time (hh:mm)	Bottles (total to lab)	Filtered (0.45 $\mu\text{m}$ )	Remarks
Purging/Sampling Device Decon Process:					

### COMMENTS:

NAVSUPPACT MID-SOUTH BUILDING CAC ACCESS / ISSUE APPLICATION				
<b>From:</b> Robert Morrison <b>To:</b> Email to <a href="mailto:MILL_BADGE@NAVY.MIL">MILL_BADGE@NAVY.MIL</a> <b>Subj:</b> Request for Identification Badge, and access to restricted spaces				
APPLICANT INFORMATION				
Name (Last, First, Middle Initial)		Gender	Citizenship	SSN:
Command/Dep. NAVFAC/PWD MIDSOUTH		Title:		Date of Birth:
Race:		State Drivers License#:		
Height:	Weight:	Hair Color:	Eye Color:	Work Phone:
Company Name: (Contractors):		Contract Exp Date:	Contract Number:	
COMMAND ACCESS REQUEST				
<input checked="" type="checkbox"/> New Access		<input type="checkbox"/> Access Modification		<input type="checkbox"/> Non-CAC Contractor
REASON FOR BADGE ISSUANCE				
<input checked="" type="checkbox"/> Initial Issue		<input type="checkbox"/> Renewal		<input type="checkbox"/> Replacement
EXTERNAL BUILDING ACCESS:				
External Building(s), Days, and Time for access(EX: 455, Mon-Fri, 0600-1800) Building 455, Mon. – Fri, 0600 - 2400				
RESTRICTED SPACE(s) ACCESS REQUIRED:				
Building(s), Room Number, Days, Time for access(EX: 769, Room 188, 24 X 7)				
ONLY COMMAND APPOINTED AUTHORIZED PERSONNEL CAN SIGN REQUEST				
Authorizing Official: (Last Name, First and Middle Initial)			Telephone Number:	
Authorizing Official Signature: (N/A when emailed, verified by email from authorizing official)			Date:	
Privacy Act Statement				
AUTHORITY: 5 U.S.C. 301; EO 12356; EO 9397 PRINCIPAL PURPOSE: To facilitate verification of a personnel security clearance for an individual applying for building access in connection with their livelihood or official duties. ROUTINE USES; Information may be furnished to Federal, state, or local agencies for regulatory and law enforcement purposes. DISCLOSURE: Voluntary; however, refusal to furnish requested information may result in inability to verify essential personal information and approve requested building pass application.				





Rev

## Field Measurement of Dissolved Oxygen

Solubility of Oxygen in Water			
at Atmospheric Pressure <sup>1,2</sup>			
Temperature	Oxygen Solubility	Temperature	Oxygen Solubility
oC	mg/L	oC	mg/L
0.0	14.621	26.0	8.113
1.0	14.216	27.0	7.968
2.0	13.829	28.0	7.827
3.0	13.460	29.0	7.691
4.0	13.107	30.0	7.559
5.0	12.770	31.0	7.430
6.0	12.447	32.0	7.305
7.0	12.139	33.0	7.183
8.0	11.843	34.0	7.065
9.0	11.559	35.0	6.950
10.0	11.288	36.0	6.837
11.0	11.027	37.0	6.727
12.0	10.777	38.0	6.620
13.0	10.537	39.0	6.515
14.0	10.306	40.0	6.412
15.0	10.084	41.0	6.312
16.0	9.870	42.0	6.213
17.0	9.665	43.0	6.116
18.0	9.467	44.0	6.021
19.0	9.276	45.0	5.927
20.0	9.092	46.0	5.835
21.0	8.915	47.0	5.744
22.0	8.743	48.0	5.654
23.0	8.578	49.0	5.565
24.0	8.418	50.0	5.477
25.0	8.263		
1. The table provides three decimals to aid interpolation			
2. Under equilibrium conditions, the partial pressure of oxygen in air-saturated water is equal to that of the oxygen in water saturated			

# Well Construction Form

Facility/Project Name:

Well ID.:

Facility License Number:

Type of Well:

Ground Water Monitoring ☐

Piezometer ☐ Injection ☐

Other \_\_\_\_\_

Date Well Installed:

Location of well relative to waste source:

Upgradient ☐ Downgradient ☐ Side-gradient ☐ Unknown ☐

Well Installed By:

Well Driller License Number:

Geologist:

A. Protective pipe: ft. above grade

B. Well casing, top elevation: ft. MSL

C. Land Surface Elevation: ft. MSL

D. Surface seal, bottom: ft. below grade

12. USCS classification of soil near screen:

GP ☐ GM ☐ GC ☐ GW ☐ SP ☐ SM ☐

SC ☐ SW ☐ ML ☐ MH ☐ CL ☐ CH ☐

Bedrock ☐

13. Sieve analysis attached? Yes ☐ No ☐

14. Drilling method used: Rotary ☐ HSA ☐

Other:

15. Drilling fluid used:

Water ☐ Air ☐ Drilling Mud ☐ None ☐

16. Drilling additives used? Yes ☐ No ☐

Specify:

17. Source of water:

E. Bentonite seal: top ft. (depth)

F. Fine sand: top ft. (depth)

G. Filter pack: top ft. (depth)

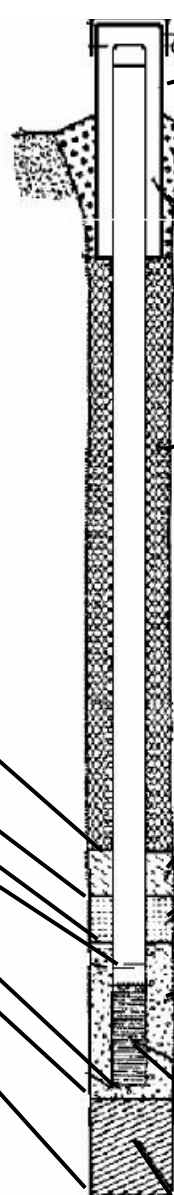
H. Screen joint top: ft. (depth)

I. Well bottom: ft. (depth)

J. Filter pack: bottom ft. (depth)

K. Borehole: bottom . ft. (depth)

Borehole diameter: in.



1. Cap and lock? ☐ Yes ☐ No

2. Protective cover pipe:

a. Inside diameter: in.

b. Length: ft.

c. Material: Steel ☐ Other

3. Surface seal: Bentonite ☐ Concrete ☐

Other:

4. Material blw. well casing and protective pipe:

Bentonite ☐ Annular space seal ☐

Other:

5. Annular space seal: (Manufacturer name)

a. Granular bentonite ☐

b. Bentonite/Cement slurry ☐

% bentonite ..... Bentonite/cement grout ☐

Lbs/gal mud weight ... bentonite slurry ☐

c. How installed: Tremie ☐ Tremie pumped ☐

Gravity ☐

6. Bentonite seal: (Manufacturer, product name)

Bentonite granules ☐

☐ 1/4 in. ☐ 3/8 in. ☐ 1/2 in. Bentonite pellets ☐

Other:

7. Fine sand material: (Manufacturer, product name, mesh size)

Volume added: ft<sup>3</sup>

8. Filter pack material: (Manufacturer, product name, mesh size)

Volume added: ft<sup>3</sup>

9. Well casing: Flush-threaded Sch 40 PVC ☐

Flush-threaded Sch 80 PVC ☐

Other:

10. Screen material:

a. Screen type: factory cut ☐ continuous slot ☐

Other:

b. Manufacturer:

c. Slot size: 0. in.

d. Slotted length: ft.

11. Backfill material: or None ☐

## CERTIFICATION:

I hereby certify that the information on this form is true and correct to the best of my knowledge:

(Signature)

(Company Name)

**Appendix C**  
**Laboratory Accreditation Certificate**



# CERTIFICATE OF ACCREDITATION

**ANSI-ASQ National Accreditation Board/AClass**  
500 Montgomery Street, Suite 625, Alexandria, VA 22314, 877-344-3044

This is to certify that

**Gulf Coast Analytical Laboratories, Inc.**  
**7979 GSRI Avenue**  
**Baton Rouge, LA 70820**

has been assessed by AClass  
and meets the requirements of

**ISO/IEC 17025:2005 and DoD-ELAP**

while demonstrating technical competence in the field(s) of

**TESTING**

Refer to the accompanying Scope(s) of Accreditation for information regarding the types of tests to which this accreditation applies.

ADE-1482

Certificate Number

AClass Approval



Certificate Valid: 08/15/2012-09/09/2014  
Version No. 003 Issued: 08/29/2012



This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2005. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated January 2009).



## ANSI-ASQ National Accreditation Board

### SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005 & DoD-ELAP

#### Gulf Coast Analytical Laboratories, Inc.

7979 GSRI Avenue, Baton Rouge, LA 70820  
Karen S. Varnado Phone: 225-769-4900

#### TESTING

Valid to: September 9, 2014

Certificate Number: ADE- 1482

#### I. Environmental

MATRIX	SPECIFIC TEST or GROUP OF ANALYTES**	SPECIFICATION OR STANDARD METHOD (all EPA unless specified)	* KEY EQUIPMENT OR TECHNOLOGY USED
Water	Flashpoint / Ignitability	1010A	Automated FP Analyzer
Water	Alkalinity	SM 2320B / 310.1	Autotitrator
Water	Acidity	SM 2310B	Autotitrator
Water / Solid	Ammonia	SM 4500 NH3 B & E, 18th ed. SM 4500 NH3 B & C, 20 <sup>th</sup> ed.	Autotitrator
Water	Mercury	7470A	CVAA
Solid	Mercury	7471B	CVAA
Water	Mercury	245.1 / 245.2	CVAA
Water / Solid	Cyanide	9012B	FIA
Water / Solid	Total Phenols	420.4 / 9066	FIA
Solid	Chloride	9251	FIA
Water / Solid	Chloride	9251 / 325.2 / SM 4500 Cl E	FIA
Water / Solid	Nitrate/Nitrite/N+N	353.2	FIA
Water / Solid	Total Phosphorous	365.1	FIA
Water / Solid	Reactive Cyanide	SW846 Sec 7.3	FIA
Water / Solid	Pesticides	8081A	GC-ECD
Water / Solid	PCB's	8082A	GC-ECD
Water / Solid	Herbicides	8151A	GC-ECD





<b>MATRIX</b>	<b>SPECIFIC TEST or GROUP OF ANALYTES**</b>	<b>SPECIFICATION OR STANDARD METHOD (all EPA unless specified)</b>	<b>* KEY EQUIPMENT OR TECHNOLOGY USED</b>
Water	EDB / DBCP	8011	GC-ECD
Water / Solid	DRO / ORO	8015B	GC-FID
Water / Solid	GRO	8015B	GC-FID
Water / Solid	TPH	TX 1005 / TX 1006	GC-FID
Water / Solid	TPH	Florida PRO	GC-FID
Water / Solid	Aromatic Volatile Organics	8021B	GC-FID/PID
Water	Dissolved Gases	RSK-175	GC-FID/TCD
Water / Solid	VOCs	8260B	GC-MS
Water / Solid	SVOCs	8270C / 8270D / SIM	GC-MS
Water	VOCs	624	GC-MS
Water	SVOCs	625	GC-MS
Water / Solid	OP Pesticides	8141A	GC-NPD
Water	TSS	SM 2540D / 160.2	Gravimetric
Water	TDS	SM 2540C / 160.1	Gravimetric
Water	TS	SM 2540B / 160.3	Gravimetric
Solid	TS	SM 2540B	Gravimetric
Water / Solid	Explosives	8330A	HPLC
Water / Solid	PAH's	8310	HPLC
Water	Anions	300.0	IC
Water / Solid	Anions	9056A	IC
Water	Perchlorate	314.0	IC
Water / Solid	ICP Metals	6010B / 6010C	ICP
Water	ICP Metals	200.7	ICP
Water	Volatile Fatty Acids	GCAL SOP WL-070	Ion Chromatography
Water / Solid	Ammonia	SM 4500 NH3 B & F 18 <sup>th</sup> ed	Ion Selective Electrode



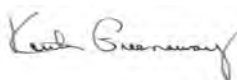
<b>MATRIX</b>	<b>SPECIFIC TEST or GROUP OF ANALYTES</b>	<b>SPECIFICATION OR STANDARD METHOD (all EPA unless specified)</b>	<b>* KEY EQUIPMENT OR TECHNOLOGY USED</b>
Water	Solid Phase Extraction	3535A	N/A
Water	Separatory Funnel Extraction	3510C	N/A
Water	Metals Digestion ICP	3010A	N/A
Solid	Ultrasonic Extraction for SVOA Analysis	3550C	N/A
Solid	Soxhlet Extraction for SVOA Analysis	3540C	N/A
Solid	Metals Digestion ICP	3050B	N/A
Water / Solid	TCLP	1311	N/A
Solid	Paint Filter Test	9095B	N/A
Water	pH	SM 4500 H+B / 9040C	pH Meter
Solid	pH	9045D	pH Meter
Water	VOC's	5030B	Purge and Trap
Solid	VOC's	5035	Purge and Trap
Water	Oil & Grease	1664A	SPE/Gravimetric
Water	Hexavalent Chromium	7196A	Spectrophotometer
Water	Sulfide	SM 4500 S2 D / 376.2	Spectrophotometer
Water	COD	HACH 8000	Spectrophotometer
Water / Solid	Sulfate	9038 / 375.4	Spectrophotometer
Water	Ortho Phosphate	SM 4500 P E	Spectrophotometer
Water	Silica	SM 4500 Si D	Spectrophotometer
Water/Solid	TKN	SM4500 Norg C SM4500 NH3 B SM4500NH3 E	Spectrophotometer
Water	Sulfide	SM 4500 S2 E / 376.1	Titration
Water / Solid	Reactive Sulfide	SW846 Sec 7.3	Titration
Solid	TOC	Lloyd Kahn 9060A	TOC Analyzer
Water	TOC	SM 5310B / 415.1 / 9060A	TOC Analyzer



MATRIX	SPECIFIC TEST or GROUP OF ANALYTES	SPECIFICATION OR STANDARD METHOD (all EPA unless specified)	* KEY EQUIPMENT OR TECHNOLOGY USED
Water	TOX	9020B	TOX Analyzer
Water	Turbidity	SM2130B	Turbidimeter
Solid	Ignitability	1030	N/A
Water	Massachusetts EPH	Massachusetts EPH	GC-FID
Solid	Massachusetts EPH	Massachusetts EPH	GC-FID
Water	Massachusetts VPH	Massachusetts VPH	GC-FID/PID
Solid	Massachusetts VPH	Massachusetts VPH	GC-FID/PID
Water/Solid	ICP-MS Metals	6020A/200.8	ICP-MS
Water	ICP-MS Metals	200.8	ICP-MS

**Notes:**

1. \* = As Applicable
2. \*\*=Refer to accredited analyte listing for exact analyte(s) in which the lab is accredited.
3. This scope is part of and must be included with the Certificate of Accreditation No. ADE- 1482



Vice President



Accredited Analytes/Methods (by matrix)														
Gulf Coast Analytical Laboratories, Inc.														
Baton Rouge, LA														
NELAC Code	Analyte	Matrix												
		Aqueous							Solid					
1000	Aluminum	6010B	6010C	6020A	200.7	200.8			6010B	6010C	6020A			
1005	Antimony	6010B	6010C	6020A	200.7	200.8	3010 A	1311	6010B	6010C	6020A		1311	6010C
1010	Arsenic	6010B	6010C	6020A	200.7	200.8	3010 A	1311	6010B	6010C	6020A		1311	6010C
1015	Barium	6010B	6010C	6020A	200.7	200.8	3010 A	1311	6010B	6010C	6020A		1311	6010C
1020	Beryllium	6010B	6010C	6020A	200.7	200.8	3010 A	1311	6010B	6010C	6020A		1311	6010C
1025	Boron	6010B	6010C		200.7				6010B	6010C				
1030	Cadmium	6010B	6010C	6020A	200.7	200.8	3010 A	1311	6010B	6010C	6020A		1311	6010C
1035	Calcium	6010B	6010C	6020A	200.7	200.8			6010B	6010C	6020A			
1040	Chromium	6010B	6010C	6020A	200.7	200.8	3010 A	1311	6010B	6010C	6020A		1311	6010C
1045	Chromium VI	7196A												
1050	Cobalt	6010B	6010C	6020A	200.7	200.8			6010B	6010C	6020A			
1055	Copper	6010B	6010C	6020A	200.7	200.8			6010B	6010C	6020A			
1070	Iron	6010B	6010C	6020A	200.7	200.8			6010B	6010C	6020A			
1075	Lead	6010B	6010C	6020A	200.7	200.8	3010 A	1311	6010B	6010C	6020A		1311	6010C
1085	Magnesium	6010B	6010C	6020A	200.7	200.8			6010B	6010C	6020A			
1090	Manganese	6010B	6010C	6020A	200.7	200.8			6010B	6010C	6020A			
1095	Mercury	7470A	245.2	245.1			3010 A	1311	7471B				1311	6010C
1100	Molybdenum	6010B	6010C	6020A	200.7	200.8			6010B	6010C	6020A			
1105	Nickel	6010B	6010C	6020A	200.7	200.8			6010B	6010C	6020A			
1125	Potassium	6010B	6010C	6020A	200.7	200.8			6010B	6010C	6020A			
1140	Selenium	6010B	6010C	6020A	200.7	200.8	3010 A	1311	6010B	6010C	6020A		1311	6010C
1150	Silver	6010B	6010C	6020A	200.7	200.8	3010 A	1311	6010B	6010C	6020A		1311	6010C
1155	Sodium	6010B	6010C	6020A	200.7	200.8			6010B	6010C	6020A			
1160	Strontium	6010B	6010C	6020A	200.7	200.8			6010B	6010C	6020A			
1165	Thallium	6010B	6010C	6020A	200.7	200.8	3010 A	1311	6010B	6010C	6020A		1311	6010C
1175	Tin	6010B	6010C	6020A	200.7	200.8			6010B	6010C	6020A			
1180	Titanium	6010B	6010C	6020A	200.7	200.8			6010B	6010C	6020A			
1185	Vanadium	6010B	6010C	6020A	200.7	200.8			6010B	6010C	6020A			
1190	Zinc	6010B	6010C	6020A	200.7	200.8	3010 A	1311	6010B	6010C	6020A		1311	6010C
1192	Zirconium			6020A		200.8					6020A			
1500	Acidity(as CaCO3)	SM 2310B												
1505	Total Alkalinity(as CaCO3)	SM 2320B	EPA 310.1											
1515	Ammonia as N	SM4500 NH3 B & E	SM4500 NH3 B & F	SM4500 NH3 B & C					SM 4500 NH3 BE	SM 4500 NH3 BF	SM4500 NH3 B & C			
1540	Bromide	9056A	EPA 300.0						9056A					
1565	COD	HACH 8000												
1575	Chloride	9056A	EPA 300.0	9251	EPA 325.2			SM 4500 Cl E	9056A	9251				
1625	Corrosivity (pH)	9040C	SM 4500 H+B						9045D					
1645	Total Cyanide	9012B							9012B					
1730	Fluoride	9056A	EPA 300.0						9056A					
1755	Total Hardness (as CaCO3)	6020A	200.8											
1780	Ignitability	1010A							1010A					
1810	Nitrate as N	9056A	EPA 300.0	EPA 353.2					9056A	EPA 353.2				
1820	Nitrate and Nitrite as N	9056A	EPA 300.0	EPA 353.2					9056A	EPA 353.2				
1840	Nitrite as N	9056A	EPA 300.0	EPA 353.2					9056A	EPA 353.2				
1860	Oil & Grease	EPA 1664A												
1870	Orthophosphate as P	SM 4500 PE												
1895	Perchlorate	EPA 314.0							EPA 314.0					
1900	pH	9040C	SM 4500 H+B						9040C					
1905	Total Phenolics (4AAP)	9066	EPA 420.4						9066	EPA 420.4				
1910	Total Phosphorous	EPA 365.1							EPA 365.1					
1925	Reactive sulfide	SW846 Sec 7.3							SW846 Sec 7.3					
-	Reactive Cyanide	SW846 Sec 7.3							SW846 Sec 7.3					
-	Percent Moisture								SM 2540G					
1950	Total Solids	SM 2540B	EPA 160.3						SM2540 G					
1955	Total Dissolved Solids at 180° (TFR)	SM 2540C	EPA 160.1											
1960	Non-Filterable Residue (TSS)	SM 2540D	EPA 160.2											
2000	Sulfate	9056A	EPA 300.0	9038	EPA 375.4				9056A	9038	9038	EPA 375.4		

Accredited Analytes/Methods (by matrix)														
Gulf Coast Analytical Laboratories, Inc.														
Baton Rouge, LA														
NELAC Code	Analyte	Matrix												
		Aqueous							Solid					
2005	Sulfide	SM 4500 S2 D	EPA 376.2	SM 4500 S2 E	EPA 376.1									
2040	TOC	SM 5310B	EPA 415.1	9060A					EPA 9060A					
2045	Total Organic Halides	9020B							9020B					
2055	Turbidity	SM 2130 B												
4315	Acetone	8260B	624						8260B					
4320	Acetonitrile	8260B	624						8260B					
4325	Acrolein	8260B	624						8260B					
4340	Acrylonitrile	8260B	624						8260B					
4375	Benzene	8260B	624	1311					8260B	1311				
4385	Bromobenzene	8260 B							8260 B					
4390	Bromochloromethane	8260B	624						8260B					
4395	Bromodichloromethane	8260B	624						8260B					
4400	Bromoform	8260B	624						8260B					
4410	2-Butanone (MEK)	8260B	624	1311					8260B	1311				
4435	n-Butylbenzene	8260B	624						8260B					
4440	sec-Butylbenzene	8260B	624						8260B					
4445	tert-Butylbenzene	8260B	624						8260B					
4450	Carbon disulfide	8260B	624						8260B					
4455	Carbon tetrachloride	8260B	624	1311					8260B	1311				
4475	Chlorobenzene	8260B	624	1311					8260B	1311				
4485	Chloroethane	8260B	624						8260B					
4500	2-Chloroethylvinylether	8260B	624						8260B					
4505	Chloroform	8260B	624	1311					8260B	1311				
4535	2-Chlorotoluene	8260B	624						8260B					
4540	4-Chlorotoluene	8260B	624						8260B					
4570	1,2-Dibromo-3-chloropropane (DBCP)	8260B	624	8011					8260B					
4575	Dibromochloromethane	8260B	624						8260B					
4585	1,2-Dibromoethane (EDB)	8260B	624	8011					8260B					
4595	Dibromomethane	8260B	624						8260B					
4610	1,2 Dichlorobenzene	8260B	624		8270C	8270D	625		8260B		8270C	8270D		
4615	1,3 Dichlorobenzene	8260B	624		8270C	8270D	625		8260B		8270C	8270D		
4620	1,4 Dichlorobenzene	8260B	624	1311	8270C	8270D	625		8260B	1311	8270C	8270D		
4625	Dichlorodifluoromethane	8260B	624						8260B					
4630	1,1-Dichloroethane	8260B	624						8260B					
4635	1,2 Dichloroethane	8260B	624	1311					8260B	1311				
4640	1,1-Dichloroethene	8260B	624	1311					8260B	1311				
4645	cis-1,2-Dichloroethene	8260B	624						8260B					
4655	1,2-Dichloropropane	8260B	624						8260B					
4660	1,3-Dichloropropane	8260B	624						8260B					
4665	2,2-Dichloropropane	8260B	624						8260B					
4670	1,1-Dichloropropene	8260B	624						8260B					
4680	cis-1,3-Dichloropropylene	8260B	624						8260B					
4685	trans-1,3-Dichloropropene	8260B	624						8260B					
4700	trans-1,2-Dichloroethene	8260B	624						8260B					
4740	p-Dioxane				8270C	8270D	625							
4765	Ethylbenzene	8260B	624						8260B					
4835	Hexachlorobutadiene	8260B	624	1311	8270C	8270D	625		8260B	1311	8270C	8270D		
4840	Hexachloroethane			1311	8270C	8270D	625			1311	8270C	8270D		
4860	2-Hexanone	8260B	624						8260B					
4900	Isopropylbenzene	8260B	624						8260B					
4910	p-Isopropyltoluene	8260B	624						8260B					
4950	Bromomethane	8260B	624						8260B					
4960	Chloromethane	8260B	624						8260B					
	Mrthyl Acetate	8260B							8260B					
4975	Methylene Chloride	8260B	624						8260B					
	Methylcyclohexane	8260B							8260B					
4995	4-Methyl-2-pentanone (MIBK)	8260B	624						8260B					

Accredited Analytes/Methods (by matrix)														
Gulf Coast Analytical Laboratories, Inc.														
Baton Rouge, LA														
NELAC Code	Analyte	Matrix												
		Aqueous							Solid					
5000	Methyl tert-butyl ether (MTBE)	8260B	624						8260B					
5005	Naphthalene	8260B	624	8310	8270 C	8270D		625	8260B		8270C	8270D		8310
5015	Nitrobenzene	8330 A		1311	8270C	8270D		625		1311	8270C	8270D		8330A
5090	n-Propylbenzene	8260B	624						8260B					
5095	Pyridine			1311	8270C	8270D		625		1311	8270C	8270D		
5100	Styrene	8260B	624						8260B					
5105	1,1,1,2-Tetrachloroethane	8260B	624						8260B					
5110	1,1,2,2-Tetrachloroethane	8260B	624						8260B					
	1,2,4,5-Tetrachlorobenzene	8260B							8260B					
5115	Tetrachloroethene	8260B	624	1311					8260B	1311				
5140	Toluene	8260B	624						8260B					
5145	o-Toluidine				8270C	8270D		625						
	1,1,2-trichloro-1,2,2-trifluoroethane	8260B							8260B					
5150	1,2,3-Trichlorobenzene	8260B	624						8260B					
5155	1,2,4-Trichlorobenzene	8260B	624		8270C	8270D		625	8260B	8270 C	8270 D			
5160	1,1,1-Trichloroethane	8260B	624						8260B					
5165	1,1,2-Trichloroethane	8260B	624						8260B					
5170	Trichloroethene	8260B	624	1311					8260B	1311				
5175	Trichlorofluoromethane	8260B	624						8260B					
5180	1,2,3-Trichloropropane	8260B	624						8260B					
5210	1,2,4-Trimethylbenzene	8260B	624						8260B					
5215	1,3,5-Trimethylbenzene	8260B	624						8260B					
5225	Vinyl acetate	8260B	624						8260B					
5235	Vinyl chloride	8260B	624	1311					8260B	1311				
5240	m+p-Xylene	8260B	624						8260B					
5250	o-Xylene	8260B	624						8260B					
5260	Xylenes, total	8260B	624						8260B					
5500	Acenaphthene	8310			8270C	8270D		625			8270C	8270D		8310
5505	Acenaphthylene	8310			8270C	8270D		625			8270C	8270D		8310
	Acetophenone				8270C						8270C			
5545	Aniline				8270C	8270D		625			8270C	8270D		
5555	Anthracene	8310			8270C	8270D		625			8270C	8270D		8310
	Benzaldehyde				8270C						8270C			
5575	Benzo(a)anthracene	8310			8270C	8270D		625			8270C	8270D		8310
5580	Benzo(a)pyrene	8310			8270C	8270D		625			8270C	8270D		8310
5585	Benzo(b)fluoranthene	8310			8270C	8270D		625			8270C	8270D		8310
5590	Benzo(g,h,i)perylene	8310			8270C	8270D		625			8270C	8270D		8310
5595	Benzidine				8270C	8270D		625			8270C	8270D		
5600	Benzo(k)fluoranthene	8310			8270C	8270D		625			8270C	8270D		8310
5610	Benzoic acid				8270C	8270D		625			8270C	8270D		
5630	Benzyl alcohol				8270C	8270D		625			8270C	8270D		
	Biphenyl				8270C						8270C			
5660	4-Bromophenyl-phenylether				8270C	8270D		625			8270C	8270D		
5670	Butyl benzyl phthalate				8270C	8270D		625			8270C	8270D		
5680	Carbazole				8270C	8270D		625			8270C	8270D		
5700	4-Chloro-3-methylphenol				8270C	8270D		625			8270C	8270D		
5745	4-Chloroaniline				8270C	8270D		625			8270C	8270D		
5760	bis(2-Chloroethoxy)methane				8270C	8270D		625			8270C	8270D		
5765	bis(2-Chloroethyl)ether				8270C	8270D		625			8270C	8270D		
5780	bis(2-Chloroisopropyl) ether				8270C	8270D		625			8270C	8270D		
5795	2-Chloronaphthalene				8270C	8270D		625			8270C	8270D		
5800	2-Chlorophenol				8270C	8270D		625			8270C	8270D		
5825	4-Chlorophenyl-phenylether				8270C	8270D		625			8270C	8270D		
5855	Chrysene	8310			8270C	8270D		625			8270C	8270D		8310
5895	Dibenzo(a,h)anthracene	8310			8270C	8270D		625			8270C	8270D		8310
5905	Dibenzofuran				8270C	8270D		625			8270C	8270D		
5925	Di-n-butylphthalate				8270C	8270D		625			8270C	8270D		



Accredited Analytes/Methods (by matrix)														
Gulf Coast Analytical Laboratories, Inc.														
Baton Rouge, LA														
NELAC Code	Analyte	Matrix												
		Aqueous							Solid					
5945	3,3'-Dichlorobenzidine				8270C	8270D		625			8270C	8270D		
6000	2,4-Dichlorophenol				8270C	8270D		625			8270C	8270D		
6005	2,6-Dichlorophenol				8270C	8270D		625			8270C	8270D		
6070	Diethyl phthalate				8270C	8270D		625			8270C	8270D		
6130	2,4-Dimethylphenol				8270C	8270D		625			8270C	8270D		
6135	Dimethyl phthalate				8270C	8270D		625			8270C	8270D		
6160	1,3-Dinitrobenzene	8330 A							8330A					
6175	2,4-Dinitrophenol				8270C	8270D		625			8270C	8270D		
6185	2,4-Dinitrotoluene	8330 A	1311		8270C	8270D		625		1311	8270C	8270D	8330A	
6190	2,6-Dinitrotoluene	8330 A			8270C	8270D		625			8270C	8270D	8330A	
6200	Di-n-octylphthalate				8270C	8270D		625			8270C	8270D		
6255	bis(2-ethylhexyl) phthalate				8270C	8270D		625			8270C	8270D		
6265	Fluoranthene	8310			8270C	8270D		625			8270C	8270D		8310
6270	Fluorene	8310			8270C	8270D		625			8270C	8270D		8310
6275	Hexachlorobenzene		1311		8270C	8270D		625		1311	8270C	8270D		
6285	Hexachlorocyclopentadiene				8270C	8270D		625			8270C	8270D		
6315	Indeno(1,2,3, cd)pyrene	8310			8270C	8270D		625			8270C	8270D		8310
6320	Isophorone				8270C	8270D		625			8270C	8270D		
6360	2-Methyl-4,6-Dinitrophenol				8270C	8270D		625			8270C	8270D		
6380	1-Methylnaphthalene				8270C	8270D		625						
6385	2-Methylnaphthalene	8310			8270C	8270D		625			8270C	8270D		8310
6400	2-Methylphenol		1311		8270C	8270D		625		1311	8270C	8270D		
6410	4-Methylphenol (and/or 3-Methylphenol)		1311		8270C	8270D		625		1311	8270C	8270D		
6415	Tetryl (Methyl-2,4,6-trinitrophenylnitramine)	8330 A							8330 A					
6460	2-Nitroaniline				8270C	8270D		625			8270C	8270D		
6465	3-Nitroaniline				8270C	8270D		625			8270C	8270D		
6470	4-Nitroaniline				8270C	8270D		625			8270C	8270D		
6490	2-Nitrophenol				8270C	8270D		625			8270C	8270D		
6500	4-Nitrophenol	8151 A			8270C	8270D		625	8151 A		8270C	8270D		
6525	N-Nitrosodiethylamine				8270C	8270D		625			8270C	8270D		
6530	N-Nitrosodimethylamine				8270C	8270D		625			8270C	8270D		
6535	N-Nitrosodiphenylamine				8270C	8270D		625			8270C	8270D		
6545	N-Nitroso-di-n-propylamine				8270C	8270D		625			8270C	8270D		
6590	Pentachlorobenzene				8270C	8270D		625			8270C	8270D		
6605	Pentachlorophenol	8151 A	1311		8270C	8270D		625	8151 A	1311	8270C	8270D		
6615	Phenanthrene	8310			8270C	8270D		625			8270C	8270D		8310
6625	Phenol				8270C	8270D		625			8270C	8270D		
6665	Pyrene	8310			8270C	8270D		625			8270C	8270D		8310
6715	1,2,4,5-Tetrachlorobenzene				8270C	8270D		625			8270C	8270D		
6735	2,3,4,6-Tetrachlorophenol				8270C	8270D		625			8270C	8270D		
6835	2,4,5-Trichlorophenol		1311		8270C	8270D		625		1311	8270C	8270D		
6840	2,4,6-Trichlorophenol		1311		8270C	8270D		625		1311	8270C	8270D		
6885	1,3,5-Trinitrobenzene	8330 A											8330A	
7025	Aldrin	8081B											8081B	
7075	Azinphos-methyl (Guthion)	8141B							8141B					
7105	delta-BHC	8081B											8081B	
7110	alpha-BHC	8081B											8081B	
7115	beta-BHC	8081B											8081B	
7120	gamma-BHC (Lindane)	8081B	1311							1311			8081B	
7240	alpha-Chlordane	8081B											8081B	
7245	gamma-Chlordane	8081B											8081B	
7250	Chlordane (total)	8081B											8081B	
7355	DDD (4,4)	8081B											8081B	
7360	DDE (4,4)	8081B											8081B	
7365	DDT (4,4)	8081B											8081B	
7410	Diazinon	8141B							8141B					
7470	Dieldrin	8081B											8081B	

Accredited Analytes/Methods (by matrix)															
Gulf Coast Analytical Laboratories, Inc.															
Baton Rouge, LA															
NELAC Code	Analyte	Matrix													
		Aqueous							Solid						
7510	Endosulfan I	8081B											8081B		
7515	Endosulfan II	8081B											8081B		
7520	Endosulfan sulfate	8081B											8081B		
7530	Endrin aldehyde	8081B											8081B		
7535	Endrin ketone	8081B											8081B		
7540	Endrin	8081B	1311							1311			8081B		
7685	Heptachlor	8081B	1311							1311			8081B		
7690	Heptachlor Epoxide (beta)	8081B	1311							1311			8081B		
7770	Malathion	8141B							8141B						
7775	MCPA	8151A							8151A						
7780	MCPP	8151 A							8151 A						
7810	Methoxychlor	8081B	1311							1311			8081B		
7825	Parathion, methyl	8141B							8141B						
7955	Parathion, ethyl	8141B							8141B						
7985	Phorate	8141B							8141B						
8110	Ronnel	8141B							8141B						
8200	Stirophos	8141B							8141B						
8250	Toxaphene (total)	8081B											8081B		
8505	Acifluorfen	8151 A							8151 A						
8530	Bentazon	8151 A							8151 A						
8540	Chloramben	8151 A							8151 A						
8545	2,4-D	8151 A	1311						8151A	1311					
8550	Dacthal (DCPA)	8151 A							8151 A						
8555	Dalapon	8151 A							8151 A						
8560	2,4-DB	8151 A							8151A						
8595	Dicamba	8151 A							8151A						
8600	3,5-Dichlorobenzoic acid	8151 A							8151 A						
8605	2,4-DP (Dichlorprop)	8151 A							8151 A						
8620	Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	8151 A							8151A						
8625	Disulfoton	8141B							8141B						
8645	Picloram	8151 A							8151 A						
8650	2,4,5-TP (Silvex)	8151 A	1311							1311			8151A		
8655	2,4,5-T	8151 A							8151A						
8880	Aroclor 1016	8082A							8082A						
8885	Aroclor 1221	8082A							8082A						
8890	Aroclor 1232	8082A							8082A						
8895	Aroclor 1242	8082A							8082A						
8900	Aroclor 1248	8082A							8082A						
8905	Aroclor 1254	8082A							8082A						
8910	Aroclor 1260	8082A							8082A						
9303	2-Amino-4,6-dinitrotoluene	8330 A												8330A	
9306	4-Amino-2,6-dinitrotoluene	8330 A												8330A	
9369	Diesel range organics (DRO)	8015C							8015C						
9432	RDX (Hexahydro-1,3,5-trinitro-1,3,5-triazine)	8330 A												8330A	
9507	2-Nitrotoluene	8330 A												8330A	
9510	3-Nitrotoluene	8330 A												8330A	
9513	4-Nitrotoluene	8330 A												8330A	
9522	HMX (Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine)	8330 A												8330A	
9651	2,4,6-Trinitrotoluene	8330 A												8330A	
	Methane	RSK-175													
	Ethane	RSK-175													
	Ethene	RSK-175													
	Carbon Dioxide	RSK-175													
	Lactic Acid	GCAL SOP WL-070													
	Formic Acid	GCAL SOP WL-070													
	Acetic Acid	GCAL SOP WL-070													
	Propionic Acid	GCAL SOP WL-070													

Accredited Analytes/Methods (by matrix)														
Gulf Coast Analytical Laboratories, Inc.														
Baton Rouge, LA														
NELAC Code	Analyte	Matrix												
		Aqueous							Solid					
	Butyric Acid	GCAL SOP WL-070												
	1-Chlorohexane	8260B												
	1-Chlorohexane								8260B					
	1,2-Diphenylhydrazine	8270C	8270D											
	1,2-Diphenylhydrazine								8270C	8270D				
	Chlordane	8082B	1311						1311	8082B				
	Toxaphene	8082 A	1311						1311	8082 A				
	Paint Filter Test	9095B							9095B					
	Oil Range Organics	8015C							8015C					
	Petroleum Hydrocarbons	Florida PRO							Florida PRO					
	Ignitability								1030					
9408	Gasoline	8015C							8015C					
4375	Benzene	8021B							8021B					
4765	Ethyl Benzene	8021B							8021B					
5140	Toluene	8021B							8021B					
5260	Xylene	8021B							8021B					
9375	DIPE	8260B							8260B					
4770	ETBE	8260B							8260B					
5000	MTBE	8260B							8260B					
4370	TAME	8260B							8260B					
4420	tert-Butyl alcohol	8260B							8260B					
9369	Diesel	8015C							8015C					
2050	Total Petroleum Hydrocarbon	TNRCC 1005							TNRCC 1005					
	Total Petroleum Hydrocarbon	TNRCC 1006							TNRCC 1006					
	GRO-Total	TNRCC 1006							TNRCC 1006					
	DRO-Total	TNRCC 1006							TNRCC 1006					
	ORO-Total	TNRCC 1006							TNRCC 1006					
	GRO-aliphatic	TNRCC 1006							TNRCC 1006					
	DRO-alphativ	TNRCC 1006							TNRCC 1006					
	ORO-aliphatic	TNRCC 1006							TNRCC 1006					
	GRO-aromatic	TNRCC 1006							TNRCC 1006					
	DRO-aromatic	TNRCC 1006							TNRCC 1006					
	ORO-aromatic	TNRCC 1006							TNRCC 1006					
-	C5-C8 Alliphatic Hydrocarbons	MADEP VPH							MADEP VPH					
-	C9-C12 Alliphatic Hydrocarbons	MADEP VPH							MADEP VPH					
-	C9-C10 Aromatic Hydrocarbons	MADEP VPH							MADEP VPH					
-	C9-C18 Alliphatic Hydrocarbons	MADEP EPH							MADEP EPH					
-	C19-C36 Alliphatic Hydrocarbons	MADEP EPH							MADEP EPH					
-	C11-C22 Aromatic Hydrocarbons	MADEP EPH							MADEP EPH					

**Appendix D**  
**Laboratory Standard Operating Procedures**

GULF COAST ANALYTICAL LABORATORIES, Inc.  
EXTRACTIONS  
STANDARD OPERATING PROCEDURES

PROCEDURE: EXT-001  
PAGE: 1 of 4  
EFFECTIVE DATE: 11/18/2011  
APPROVED BY:  
QA/QC APPROVED:

SUBJECT

SCOPE AND APPLICATION

This procedure is designed for the preparation of soil/sediment samples that may contain Base/Neutral/Acid compounds at levels of approximately 330 µg/kg to 20,000 µg/kg.

A 30 g portion of sample is mixed with anhydrous Sodium sulfate and extracted with 1:1 Methylene chloride-Acetone using an ultrasonic probe. If peaks are present at greater than 20,000 µg/kg, discard the extract and prepare the sample by the medium level method. This fraction is for GC/MS analysis of BNAs.

MATRIX

Solid

REFERENCE

SW-846 Method 3550C

PRESERVATIVE

Cool ≤ 6°C but not frozen

SAMPLE COLLECTION

Samples should be collected and stored in glass containers.

HOLDING TIME

Samples must be extracted within 14 days of sample collection. Extracts must be analyzed within 40 days following extraction.

DEFINITIONS

See Appendix A Glossary

1.625 Spiking Solution - for this laboratory, this refers to a spiking solution that contains the full list of compounds. The laboratory refers to this as the long list spiking solution.

2.8270 Spiking Solution - for this laboratory, this refers to a spiking solution that contains fewer compounds. The laboratory refers to this as the short list spiking solution. This spiking solution must not be used when analyzing samples for DOD, AFCEE, South Carolina or when full list spikes are required.

SAFETY

Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO the use of any chemicals. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn.

In addition to other measures prescribed by the division, solvents must be handled in ventilated hoods.

#### APPARATUS

250 ml beaker

Kuderna-Danish (K-D) apparatus

- A) Concentrator tube, 10 mL graduated
- B) Evaporation flask, 500 mL (attach to tube with blue clamp)
- C) Snyder column - 3 ball macro, rinsed three times with methylene chloride

Snyder column - 2 ball micro, rinsed three times with methylene chloride

Sonicator, Fisher Scientific Ultrasonic Dismembrator equipped with  $\frac{3}{4}$  inch disruptor horn.

Glass funnels - rinsed with methylene chloride

Filter paper - Fisherbrand P8, 18.5 cm or equivalent

Analytical balance, capable of weighing 0.1 g

1.0 mL pipettor or 1.0 mL gastight syringe

1.0 mL Class "A" volumetric flask

5.0 mL Class "A" volumetric flask

10.0 mL Class "A" volumetric flask

1000  $\mu$ L Eppendorf

Boiling chips - Teflon or equivalent

Pasteur Pipets and bulb

2 mL glass vials with PTFE (Teflon) lined crimp caps

Spatula, stainless steel

Hot plate

Steam bath - Organomation S-Evap-KD Solvent Evaporator, or equivalent, heated with concentric ring cover and capable of temperature control ( $\pm 5^{\circ}\text{C}$ ). The bath must be used in a hood.

#### REAGENTS

All organic solvent shall be of pesticide grade or equivalent. Label all containers and squeeze bottles with reagent ID, lot, and expiration date. Follow manufacturer's instruction for reagent expiration and storage. When the Manufacturer fails to provide an expiration date, the expiration date will be 12 months from the date opened or sooner if the reagents show signs of deterioration such as change in color, clumping, etc.

Methylene chloride

Acetone

Sodium sulfate - reagent grade, granular, anhydrous, heat to  $400^{\circ}\text{C}$  for 4 hours. Store prepared Sodium sulfate in glass jars with Teflon lined screw caps. Keep covered at all times when not in use. Record the preparation in the logbook. Label jar with prepared lot number and expiration date.

#### STANDARDS

All standards used must be pure material or from prepared certified solutions. The Certificate of Analysis shall be kept on file. Follow manufacturer's instruction for standard expiration and storage. Label all working



standards with date prepared, concentration, Standard ID number, expiration date and the initials of individual preparing the standard.

#### 1. Surrogate Standard Spiking Solution

Surrogate standards are added to all samples including QC.

Stock surrogate standard - a purchased standard prepared in methanol containing the following surrogates at the listed concentration:

50 µg/mL	100 µg/mL
Nitrobenzene-d <sub>5</sub>	Phenol-d <sub>5</sub>
2-Fluorobiphenyl	2-Fluorophenol
Terphenyl-d <sub>14</sub>	2,4,6-Tribromophenol

Store the spiking solutions at ≤ 6°C in Teflon-sealed amber glass containers. The solutions must be replaced when expired (manufacturer's date), or sooner if comparison with quality control check samples indicate a problem.

#### 2. Spiking Solutions (8270C/625) - stored at ≤ 6°C in Teflon-sealed amber glass containers until manufacturer's expiration date.

A. 625 Spiking Solution - full list of compounds. The spiking solution is purchased at a concentration of 100 µg/mL in Methanol. Spike 1.0 mL of the 625 spiking solution into the LCS/LCSD and MS/MSD.

B. Additional spiking solutions may be purchased to include additional analytes requested by the client.

### PROCEDURE

#### Sample Extraction

1. Label each beaker with the sample ID of the sample or QC sample to be extracted. Complete the extraction sheet with the sample ID's. Fill in the extraction sheet information as completed. An extraction sheet is attached.
2. With each extraction batch, a method blank, LCS/ LCSD and MS/MSD are included. Document on the extraction sheet if insufficient sample is available for a MS/MSD. For the method blank, LCS, and LCSD weigh 30 grams Sodium sulfate into a 250 mL beaker. Spike the LCS/LCSD with 1.0 mL of the appropriate spiking solution. Spike the blank and LCS/LCSD with 1.0 mL of surrogate standard spiking solution.
3. Homogenize sample by pouring the contents of the sample container onto a piece of butcher paper and chop and mix with a spatula. Remove any foreign object such as sticks, leaves, or rocks. Weigh approximately 30 g of homogenized sample to the nearest 0.1 g into a 250 mL beaker. Add 1.0 mL of the surrogate standard spiking solution to each sample and the QC samples.

Add 1.0 mL of the appropriate spiking solution to the MS/MSD. For samples requiring SIMS analysis, add 100  $\mu$ L of the surrogate standard spiking solution to each of the samples (including the QC samples) and 100  $\mu$ L of the 625 spiking solution to the LCS/LCSD and MS/MSD pairs instead of the 1.0 mL used for routine extraction of BNA samples. Add 30 g of Anhydrous Sodium sulfate. Mix well. The sample should have a free flowing sandy texture at this point. If not free flowing, add sodium sulfate until the sample is free flowing.

4. Immediately, add at least 100 mL of 1:1 Methylene Chloride/Acetone to each beaker. The solvent level should be about 2 inches above the sample in the beaker.
5. Fisher Scientific (Model 500) Dismembrator must be tested before use. Press test button on front panel. If the sonicator is functioning correctly the instrument returns to ready mode and is ready for use. If an error message is given see instrument manual. Place the probe of the sonicator about  $\frac{1}{2}$  inch below the surface of the solvent but above the sediment layer. Sonicate each sample for 3 minutes.
6. Decant and filter extracts through glass funnels lined with folded FisherBrand P8 filter paper into a KD apparatus consisting of 10 mL concentrator tube and a 500 ml evaporation flask.
7. Repeat extraction two more times with two additional 100 mL portions of 1:1 Methylene chloride-Acetone. Decant the extraction solvent after each sonication. On the final sonication, filter the entire sample and rinse filter with Methylene chloride.
8. Add 1 or 2 boiling chips. Attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1.0 mL methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10 to 15 minutes. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume is below 5 mL, remove the K-D apparatus and allow it to cool and drain for at least 10 minutes. After extract cools it will condense in the bottom of the receiver causing an increase in volume.
9. Remove the 3 ball Snyder column and evaporation flask. Add one or two clean boiling chips to the concentrator tube and attach a two-ball micro-Snyder column. Pre-wet the Snyder column by adding about 0.4 mL of methylene chloride to the top of the column. Place the concentrator tube with the micro Snyder column in a hot water bath (80 to 90°) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration

in five to 10 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches about 0.5 mL, remove the concentrator tube with the Snyder column from the water bath and allow it to cool and drain for at least 10 minutes. Adjust the final volume to 1.0 mL using a Class "A" volumetric flask with methylene chloride. Transfer the extract to an auto-sampler vial, seal with a Teflon lined crimp cap and label.

10. Enter the prep information in the LIMS. Deliver a copy of the extraction log and the extracts to the GCMS-Semivolatiles laboratory. If no one is available to receive the extracts, place the extracts in the appropriate refrigerator.

METHOD  
PERFORMANCE

1. The recovery of surrogates is used to monitor unusual matrix effects, sample processing problems, etc. The recovery of matrix spiking compounds, when compared to laboratory control sample (LCS) recoveries, indicates the presence or absence of unusual matrix effects.
2. The performance of each 3500 method will be dictated by the overall performance of the sample preparation in combination with the cleanup method and/or the analytical determinative method.

POLLUTION PREVENTION      See QAPP Section 13.2

WASTE MANAGEMENT        See SOP GEN-009

METHOD  
MODIFICATION

1. Procedure step 4 - The extraction solvent is added to the sample to be approximately 2 inches above the solid sample in the beaker. Exactly 100mL is not measured as stated in the method.
2. surrogate and matrix spikes in lieu of a syringe.

## APPENDIX A

### GLOSSARY

ACCEPTANCE LIMITS - data quality limits specified by the test method or generated by the laboratory. Specified limits placed on an item, process, or service defined in the requirement document.

ACCURACY - a measure of how close a measured value is to a known true value. Accuracy is assessed by means of reference samples and percent recoveries of spiked samples. The degree of agreement between an observed value and an accepted reference value. Accuracy includes combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

ALIQOT - a discrete, measure, or representative portion of a sample taken for analysis.

ANALYTE - the chemical element or compound an analyst seeks to determine; the chemical element of interest.

ANALYTICAL BATCH - the basic unit for analytical quality control, defined as samples that are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. Samples in each batch should be of similar composition (e.g., groundwater, sludge, ash).

ANALYTICAL SAMPLE - any solution or media introduced into an instrument on which an analysis is performed, excluding instrument calibration, initial calibration verification, initial calibration blank, continuing calibration verification, and continuing calibration blank. The following are all analytical samples: undiluted and diluted samples (EPA and non-EPA), predigestion spike samples, duplicate samples, serial dilution samples, analytical spike samples, post digestion spike samples, interference check samples (ICS), CRDL standard for AA(CRA), CRDL standard for ICP(CRI), laboratory control sample(LCS), preparation blank(PB), and linear range analysis sample(LRS).

ANALYST - the designated individual who performs the "hands on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

AREA UNITS - a term used in gas chromatography that indicates the peak area of a compound exiting a chromatographic column. The size or area of the peak is proportional to the amount of analyte in the sample.

ATOMIC ABSORPTION (AA) - a technique for analyzing metal using an element-specific lamp that emits a characteristic light spectrum. A sample is heated in a flame or graphite furnace and the light beam is passed through it. When the sample absorbs light, an energy loss is detected and is translated into a concentration of metal in the sample. This technique detects one metal at a time.

AUDIT - a systematic check to determine the quality of some function or activity. Two basic types are performance audits and system audits. Performance audits involve a quantitative comparison of the laboratory's results to those of a proficiency sample containing known concentration of analytes. A system audit is a qualitative evaluation that normally consists of an onsite review of a lab's quality assurance system and physical facilities.

BACKGROUND CORRECTION - a technique usually employed relative to metals analysis, which compensates for variable background contribution to the instrument signal in the determination of trace elements.

BATCH - environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A batch is composed of a maximum of twenty environmental samples. A preparation batch is composed of environmental sample of the same matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) and/or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices.

BIAS - the deviation of the mean of replicate analytical measurements from a reference analyte concentration. Relative bias is represented by analytical measurement mean minus the reference analyte concentration and the difference divided by the reference analyte concentration. See accuracy and precision.

4-BROMO-FLUOROBENZENE (BFB) - the compound used to check the tuning of the instrument, and is used as a surrogate compound spiked into every sample for volatile analysis. A compound utilized in EPA gas chromatography/mass spectrometry (GC/MS) volatile methods to establish mass spectral instrument performance.

BIOCHEMICAL OXYGEN DEMAND (BOD) - A measure of the amount of oxygen consumed in the biochemical processes that break down organic matter in water. A larger BOD value indicates a greater degree of organic pollution. A related term, BOD<sub>5</sub>, is the amount of dissolved oxygen consumed in five days.

BIOSEED - the bacterial culture used to inoculate a sample for testing.

BLANK - an artificial sample designed to monitor the introduction of artifacts into the measurement process. For aqueous samples, reagent water is used as a blank matrix. There are several types of blanks, which monitor a variety of processes: 1.) *laboratory blank* is taken through sample preparation and analysis only. It is a test for contamination in sample preparation and analyses. 2.) *storage blank* is stored and analyzed with samples at the laboratory. It is a test for contamination in sample storage as well as sample preparation and analysis. 3.) *trip blank* is shipped to and from the field with the sample containers. It is not opened in the field and, therefore, provides a test for contamination from sample preservation, site conditions, and transport as well as sample storage, preparation, and analysis. It is most commonly used for volatile organics. 4.) *field blank* is opened in the field and tests for contamination from the atmosphere as well as those activities listed under the trip blank.

BNA - base, neutral and acid extractable compounds. The term base, neutral and acid refer to the pH condition of the sample undergoing extraction. Certain compounds extract more efficiently from water under acidic conditions. Such compounds are often referred to as "acid extractables."

CALIBRATE - to determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device, or the correct value for each setting of a control knob. The levels of the applied calibration standard should bracket the range of planned or expect sample measurements.

CALIBRATION - The systematic determination of the relationship of the response of the measurement system to the concentration of the analyte of interest.

Instrument calibration performed before any samples are analyzed is called the initial calibration. Subsequent checks on the instrument calibration performed throughout analysis are called continuing calibration verification.

CALIBRATION BLANK (CB) - a volume of reagent water in the same matrix as the calibration standards but without the analyte.

CALIBRATION CURVE - the graphical relationship between the known values, such as concentrations, of a series of calibration standards and their analytical response.

CALIBRATION METHOD - a defined technical procedure for performing a calibration.

CALIBRATION STANDARD - a certified material used to calibrate an instrument.

CERTIFIED REFERENCE MATERIAL (CRM) - reference material, one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30)

CONTINUING CALIBRATION VERIFICATION (CCV) - used to evaluate the performance of the instrument system with respect to a defined set of method criteria. The CCVs are used to verify instrument performance during analysis. The CCV shall be prepared from the same stock standard solution used to prepare the calibration curve.

CHAIN-OF-CUSTODY-Procedures and associated documents designed to trace the custody of a sample from the point of origin to final disposition, with the intent of legally demonstrating that custody remained intact and the tampering or substitutions were precluded.

CHEMICAL OXYGEN DEMAND (COD) - A measure of the oxygen required to oxidize all compounds in water, both organic and inorganic.

COMBINED STANDARD UNCERTAINTY - The standard uncertainty of the analytical measurement result that is the sum in quadrature (square-root-of-the-sum-of-the-squares) of the component standard uncertainties.

CORRECTIVE ACTION - the action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation to prevent reoccurrence.

COVERAGE FACTOR - the numerical factor used as a multiplier of the combined standard uncertainty to expand the uncertainty corresponding to a specific level of confidence. The Student's *t*-distribution is used for determining the coverage factor.

DEMONSTRATION OF CAPABILITY (DOC) - a procedure used to establish the ability of the analyst to generate acceptable accuracy.

DEIONIZED WATER (DI) - water from which the majority of ions have been removed. Water that is contaminant free and used in the laboratory, the resistivity should be  $\leq 18$  mega ohms.

DISSOLVED METALS -- metallic elements determined on a water sample that has been passed through a 0.45-um filter.

DISSOLVED OXYGEN (DO) - the oxygen freely available in water, an indicator of water quality.

DISSOLVED SOLIDS - disintegrated organic and inorganic material contained in water. Excessive amounts make water unfit to drink or use in industrial processes.



DRY WEIGHT - the weight of a sample based on percent solids. The weight of a sample after drying in an oven at a specified temperature.

DUPLICATE MEASUREMENT - a duplicate measurement is a second measurement made on the same or identical sample of material to assist in the evaluation of measurement of variance.

DUPLICATE SAMPLE - two samples taken from the same population and carried through certain stages of sampling and testing. Duplicate sample include field co-located duplicate samples, field-split duplicate samples, and laboratory duplicate subsamples.

EXPANDED UNCERTAINTY - the quantity defining an interval enveloping the analytical measurement that captures a large fraction of the distribution of analyte concentrations that could be attributable to the quantity measured. The combined standard uncertainty is multiplied by the coverage factor to calculate the expanded uncertainty.

EXTERNAL STANDARDS - a method of quantifying chromatographic data in which standards of known concentration are analyzed prior to unknown samples. The chromatographic peak area (or height) of a sample component is compared to a calibration curve of a peak area constructed from the standard data for that component. This comparison allows the concentration of the component in the sample to be determined.

FECAL COLIFORM BACTERIA - bacteria found in mammals' intestinal tracts. Their presence in water or sludge is an indicator of pollution and possible contamination by pathogens.

FLAME IONIZATION DETECTOR (FID) - a gas chromatography detector in which the column effluent gas is mixed with hydrogen and burned in air or oxygen. The ions and electrons produced in the flame generate an electric current proportional to the amount of materials in the detector. The FID responds to nearly all organic compounds, but it does not respond to air and water, which makes it exceptionally suited to environmental analysis.

FLASHPOINT - the lowest temperature at which a flammable liquid gives off sufficient vapor to form an ignitable mixture with air near its surface or within a vessel. Combustion does not continue.

GAS CHROMATOGRAPHY (GC) - a technique for detecting organic compounds by using their physical and chemical properties to separate a mixture. The compounds are identified and quantified with various types of detectors as they exit the chromatograph. Selection of detectors is dependent on the particular compounds of interest.

GAS CHROMATOGRAPHY MASS SPECTROMETRY (GCMS) - a technique in which sample analytes are bombarded with electrons as they exit a gas chromatography column and are fragmented into characteristic ion patterns. The mass spectrometer is the detector. It can determine which fragments are present and therefore the identity of the compounds.

GLUCOSE GLUTAMIC ACID (GGA) - used as a laboratory control standard in BOD procedures.

GRAPHITE FURNACE - a technique used for the analysis of metals. An atomic absorption spectrophotometer heats the sample within a graphite tube using an electrical current. It is also commonly called a flameless furnace and

generally provides greater sensitivity for certain metals than flame or inductively coupled argon plasma techniques.

GRAVIMETRIC - analyses based on the direct or indirect weighing of the analyte in question. This technique usually requires the use of an analytical balance with a sensitivity of 0.1 mg or better.

HALL ELECTROLYTIC CONDUCTIVITY DETECTOR - an element-selective gas chromatography detector primarily intended for trace analysis of organic compounds containing chlorine, nitrogen or sulfur. In operation, this detector pyrolyzes the column effluent gas into soluble electrolytes that are dissolved in a stream of deionized liquid. The observed change in electrical conductivity, proportional to the amount of material present, is measured.

HAZARDOUS WASTE - waste regulated under RCRA that can pose a substantial or potential hazard to human health or the environment when improperly managed. Such wastes possess at least one of four characteristics (ignitability, corrosivity, reactivity, or toxicity) or appear on special EPA hazardous waste lists. The term is not interchangeable with hazardous substances or material.

HEADSPACE - Any area in a container not completely filled by the sample in which gases can collect.

HEAVY METALS - metallic elements with high atomic weights, i.e., mercury, chromium, cadmium, arsenic, and lead. They can damage the health of plants and animals at low concentrations and tend to accumulate in the food chain.

HOLDING TIME - the maximum times that samples may be held before analysis and still be considered valid or not compromised. The storage time allowed between sample collection and sample analysis when the designated preservation and storage techniques are employed.

HYDROCARBONS - chemical compounds that consist entirely of carbon and hydrogen.

ICP - Inductively coupled plasma is a type of instrument used for metals analysis. Because the temperature of the plasma is considerably higher (10,000°K) than the temperature of a flame atomic absorption spectrophotometer, it is especially useful for refractory metals. Some instruments are also capable of performing simultaneous multielement analysis.

ICP-MS - Inductively coupled plasma mass spectrometry (ICP-MS) is a type of mass spectrometry highly sensitive and capable of the determination of a range of metals and several non-metals at concentrations below one part in 10<sup>12</sup> (part per trillion). It is based on coupling together an inductively coupled plasma as a method of producing ions with a mass spectrometer as a method of separating and detecting the ions. In trace elemental analysis, the method has advantages of high speed, precision and sensitivity.

IGNITABLE - capable of burning or causing a fire.

INORGANIC CHEMICALS - chemical substances of mineral origin, unlike organic chemicals whose structure relies on carbon atoms.

INITIAL CALIBRATION VERIFICATION (ICV) - also referred to as the Independent Calibration Verification for this laboratory. A standard solution used to verify the calibration curve derived from a source independent of the instrument calibration standard. The ICV is used to quantify second source standard variance and bias.

INSTRUMENT CALIBRATION STANDARD - a reference material used to standardize an analytical instrument.

INSTRUMENT DETECTION LIMIT (IDL) - a term utilized in the EPA Inorganic Contract Laboratory Program. The IDL is three times the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration of three to five times that of the IDL on three nonconsecutive days with seven consecutive measurements per day. The IDL is the concentration equivalent to a signal, due to the analyte of interest, which is the smallest signal that can be distinguished from background noise by a particular instrument. The IDL should always be below the method detection limit, and is not used for compliance data reporting, but may be used for statistical data analysis and comparing the attributes of different instruments.

INSTRUMENT PERFORMANCE CHECK - The analyses of one of the ICSs to verified initial and continuing calibration (CCV). The IPC is used to quantify the instrumental testing repeatability variance and bias.

INSTRUMENT TUNING - a technique used in gas chromatography/mass spectrometry procedures to verify that the instrument is properly calibrated to produce reliable mass spectral information. See 4-Bromofluorobenzene under "B".

INTERNAL STANDARDS - compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for volatile organics), and sample extract (for semivolatiles) at a known concentration, prior to analysis. Internal standards are used as the basis for quantification of the target compound.

IONIZATION - Utilized in mass spectrometry to fragment analyte molecules into smaller segments. These smaller mass segments are then separated and plotted to form a "mass spectrum" which is used to identify the parent molecule. Electron impact is one example of ionization used in mass spectrometry. In more technical terms. Ionization is the process by which neutral atoms or groups of atoms become electrically charged, either positively or negatively, by the loss or gain of electrons.

ISOMERS - chemical compounds with the same molecular weight and atomic composition by differing molecular structure, e.g., n-pentane and 2-methylbutane.

LABORATORY CONTROL SAMPLE (LCS) - a clean-matrix reference sample spiked with a standard with an established analyte concentration. The LCS is carried through the entire chemical preparation and testing procedures. The LCS is used to quantify the variance and bias of the chemical preparation and instrumental testing stages without matrix interference. Also called a Laboratory fortified blank (LFB).

LABORATORY CONTROL SAMPLE DUPLICATE (LCSD) - a duplicate of the laboratory control sample in a clean matrix that is carried through the entire chemical preparation and testing procedures.

LABORATORY DUPLICATE SAMPLE (LAB DUPLICATE) - a portion of the collected sample that is carried through the chemical preparation and testing. The laboratory duplicate subsample is used to quantify the variance of the chemical preparation and instrumental testing stages with matrix interferences.

LEACHATE - a liquid that results from water collecting contaminants as it trickles through wastes, agricultural pesticides or fertilizers. Leaching may occur in farming areas, feedlots, and landfills, and may result in hazardous substances entering surface water, groundwater or soil.

LDEQ - Louisiana Department of Environmental Quality

LIBRARY SEARCH - a technique in which an unknown mass spectrum of a compound is compared to the mass spectra of compounds contained in a computer library in an

effort to identify the compound. Compounds identified in this manner are referred to as tentatively identified compounds (TICs).

LIMIT OF DETECTION (LOD) - an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent.

LIMIT OF QUANTIFICATION (LOQ) - the minimum levels, concentrations, or quantities of a target variable (e.g. target analyte) that can be reported with a specified degree of confidence..

LIMS - laboratory information management system. Horizons is the LIMS used by GCAL.

LINEAR CALIBRATION RANGE - the concentration range over which the instrument response is linear.

LOG-IN - the receipt and initial management of an environmental sample. It generally includes identifying who sent the sample; maintaining chain-of-custody; checking report and invoice information; recording analysis requested, including methodology and special instructions; and assigning a discreet in-lab identification, usually a number or bar code.

MASS SPECTRUM - a plot of ion mass/charge ratio versus intensity. A fragmentation pattern results from the particles whose mass distribution is characteristic of the parent molecule. Qualitative information is provided by a mass spectrum.

MATERIAL SAFETY DATA SHEET (MSDS) - a compilation of information required under the OSHA Communication Standard on the identity of hazardous chemicals and their associated health and physical hazards, exposure limits and precautions.

MATRIX - The physical characteristics or state of a sample - e.g., water, soil, sludge.

MATRIX INTERFERENCE - the influence of the sample matrix or sample components upon the ability of qualitatively identifies and quantitatively measures compounds in environmental samples.

MATRIX MODIFIERS - chemicals added to samples for metals analysis, which are used to lessen the effects of chemical interference, viscosity and surface tension.

MATRIX SPIKE (MS) - aliquot of a sample fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery of the spike. Matrix spiked samples are used to quantify the variance and bias of the chemical preparation and testing stages with matrix interference.

MATRIX SPIKE DUPLICATE (MSD) - a second aliquot of the same matrix as the matrix spike that is spiked to determine the precision of the method.

METHOD DETECTION LIMIT (MDL) - the minimum concentration of compound that can be measured and reported within 99% confidence that the value is above zero and is determined from analysis of a sample in a given matrix containing the analyte.

MUST - denotes a mandatory requirement.

NARRATIVE - in an analytical report, a descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

NUTRIENT - any substance assimilated by living things that promote growth. The term is generally applied to nitrogen and phosphorus in wastewater, but is also applied to other essential and trace elements.

ORGANIC - generally, any compound that contains carbon bonded to a hydrogen or halogen atom.

OXIDATION - the process in chemistry whereby electrons are removed from a molecule.

PCBs - Polychlorinated biphenyl, a group of toxic persistent chemicals used in transformers and capacitors for insulating purposes and in gas pipeline systems as a lubricant. Sale of PCBs for new uses was banned by law in 1979.

PERCENT RECOVERY - a measure of accuracy that is calculated as the measured value relative to the true value expressed as a percent.

PERFORMANCE AUDIT - a quantitative evaluation of a measurement system that involves the analysis of standard reference samples or materials which are certified as to their chemical composition or physical characteristics.

PROFICIENCY TEST (PT) SAMPLE - a sample of known composition (unknown to the laboratory), provided by an external source, which is used to evaluate lab performance. For certification purposes the PT sample is purchased from an approved provider.

pH - a numerical designation of relative acidity and alkalinity. A pH of 7.0 indicates precise neutrality. Progressively higher values indicate increasing alkalinity and lower values increasing acidity.

POLLUTANT - generally, any substance introduced into the environment that adversely affects the usefulness of a resource.

PRACTICAL QUANTITATION LIMIT (PQL) - the lowest level that can be reliably achieved within specified limits of precision and accuracy routine laboratory operating conditions. The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value.

PRECISION - a measure of the ability to reproduce analytical results. It is generally determined through the analysis of duplicate samples. The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range in either absolute or relative terms.

PRESERVATIVE - a chemical or reagent added to a sample to prevent or slow decomposition or degradation of a target analyte or a physical process. Physical and chemical preservation may be used in tandem to prevent sample deterioration.

PURGE AND TRAP - a technique used in the analysis of volatile organic where analytes are purged from a sample by means of an inert gas and trapped on a sorbent column. The sorbent is then flashheated and the analytes are transferred onto a gas chromatographic column for separation and identification.

PURGEABLE ORGANIC - an organic compound that is generally less than 20% soluble in water and has a boiling point at or below 200°C. A volatile organic. An organic compound is generally considered to be purgeable if it can be removed from water using the purging process.

QC BASED NESTED APPROACH - a procedure for estimating the uncertainty of measurement using quality control data. The procedure is described in SOP QA-013.

QUALITY ASSURANCE (QA) - all those planned and systematic actions necessary to provide adequate confidence in laboratory results. An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets the defined standards of quality with a stated level of confidence.

QUALITY ASSURANCE PROGRAM PLAN - a written assembly of management policies, objectives, principle and general procedures that outlines how the laboratory intends to generate data of known and accepted quality.

QUALITY CONTROL (QC) - the overall system of technical activities whose purpose to measure and control the quality of a product or service so that it meets the needs of the users.

QUALITY CONTROL CHART - a graph of analytical measurement results for a specific QC standard plotted sequentially with upper and lower control limits ( $\pm 3SD$ ). A central line that is the best estimate of the average variable plotted, and upper and lower marginal exceedence limits ( $\pm 4SD$ ) are usually included in the Quality Control Chart. The chart may also include upper and lower warning limits ( $\pm 2SD$ ).

QUALITY SYSTEM - a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items) and services. The quality system provides the framework for planning, implement and assessing work performed by the organization and for carrying out required QA and QC.

$r^2$  - Correlation Coefficient Squared - parameter used to evaluate the fit of linear or quadratic regression curve fit of initial calibration data.

RAW DATA - any original information from a measurement activity or study recorded in a laboratory note book, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfilm copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

REACTIVITY - the tendency of a chemical to explode under normal management conditions, to react violently when mixed with water, or to generate toxic gases.

REAGENT WATER - water in which an interference is not observed at or above the minimum quantitation limit of interest.

REFERENCE MATERIAL - a traceable standard with an established analyte concentration. Material or substance whose property values are sufficiently homogenous, stable and well established to be used for the calibration of an apparatus, the assessment of a measurement method or for assigning values to materials (ISO Guide 30)

REFERENCE METHOD - a method of known and documented accuracy and precision issued by an organization recognized as competent to do so.

RELATIVE RETENTION TIME - a measure of the shift in retention time of an analyte when referenced to an internal standard.



RELATIVE RESPONSE FACTOR (RRF) - a measure of the relative mass spectral response of an analyte compared to its internal standard. RRFs are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

REPLICATE ANALYSES - two or more results representing the same sample parameter. Replicate analyses are used to quantify the analytical measurement repeatability precision.

RPD - Relative Percent Difference, a relative difference between two analyses calculated by dividing the absolute difference of those two results by the average of their two values, then multiplying by 100.

RESOLUTION - the degree of separation between peaks eluting from a chromatographic column. Sufficient resolution between peaks is required for proper quantitation of unknown analytes.

RETENTION TIME - a term used in gas and liquid chromatography describing the time elapsed from the sample injection until the specific compound elutes or exits the chromatographic column at the detector. Each compound has a characteristic retention time on a specific column; therefore, this information is used to qualitatively identify the compounds in the sample.

SAMPLE - portion of material collected for chemical analysis, identified by a unique number assigned by the LIMS.

SHALL - denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled.

SHOULD - denotes a guideline or recommendation whenever noncompliance with the specification is permissible.

SOLID WASTE - non-liquid, non-soluble materials, ranging from municipal garbage to industrial waste, that contain complex, and sometimes hazardous, substances. Solid wastes include sewage sludge, agricultural refuse, demolition wastes, mining residues, and even liquids and gases in containers.

SOLVENT - a substance, usually liquid, capable of dissolving or dispersing one or more other substances.

SPIKE - a known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

STANDARD CURVE - a curve that plots concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by diluting the stock analyte solution in graduated amounts that cover the expected range of the samples being analyzed. The calibration standards must be prepared by using the same type of acid or solvent at the same concentration as for the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

STANDARD OPERATING PROCEDURE - a detailed written description of how a laboratory executes a particular procedure or method intended to standardize its performance.

STANDARDIZED REFERENCE MATERIAL (SRM) - a certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method.

STANDARD UNCERTAINTY - the analytical measurement uncertainty expressed as a standard deviation. The relative standard deviation represents the relative standard uncertainty.

SURROGATE - an organic compound similar to the analyte of interest in chemical composition, extraction and chromatography, but not normally found in environmental samples. Primarily used in chromatography techniques, the surrogate is spiked into quality control blanks, calibration and check standards, samples (including duplicated and QC reference samples) and spiked samples before analysis. A percent recovery is calculated for each surrogate.

SUSPENDED SOLIDS - small pollutant particles that float on the surface of, or are suspended in, sewage or other liquids. They resist removal by conventional means.

TARGET COMPOUND - specific compounds that are to be quantified in a sample based on a standard list of potential compounds.

TENTATIVELY IDENTIFIED COMPOUNDS (TICs) - compounds detected in samples that are not target compounds, internal standards, system monitoring compounds or surrogates. TICs usually consist of up to 30 peaks that are greater than 10% of the peak areas, or heights, of the nearest internal standard. They are subjected to mass spectral library searches for tentative identification. A client may specify the number of unknown peaks in its samples it wishes the laboratory to tentatively identify.

TOTAL METALS - metallic elements that have been digested prior to analysis.

TYPE A EVALUATION UNCERTAINTY - the method of evaluation of uncertainty by the statistical analysis of a series of test results.

TYPE B EVALUATION OF UNCERTAINTY - the method of evaluation of uncertainty by means other than statistical analysis.

UNCERTAINTY - the parameter associated with the analytical measurement results that characterizes the dispersion of the values that could be reasonable attributed to the quantity measured.

UNCERTAINTY INTERVAL - the range of analyte concentrations that an analytical measurement could represent at a specified level of confidence. The relative standard deviation is used to represent the relative standard uncertainty in the QC-based Nested Approach.

VALIDATION - the process for evaluating the completeness, correctness, and conformance/compliance of a specific data set against the method, procedural, or contractual specifications. It essentially evaluates performance against predetermined specifications.

VOA - volatile organic analysis

VOA BOTTLE - a vial used to contain samples for volatile organic analysis.

VOLATILE COMPOUNDS - compounds amenable to analysis by purge and trap. Synonymous with purgeable compounds.

VOLATILE ORGANIC COMPOUND (VOC) - any organic compound that participates in atmospheric photochemical reactions, except for those designated by the EPA Administrator as having negligible photochemical reactivity.

WET CHEMISTRY - procedures that involve distillations, colorimetric determinations and titrimetric measurements. Examples are chloride, nitrates, sulfates and biochemical oxygen demand.



GULF COAST ANALYTICAL LABORATORIES, INC  
EXTRACTIONS  
STANDARD OPERATING PROCEDURES

PROCEDURE: EXT-003  
PAGE: 1 of 6  
EFFECTIVE DATE: 11/18/2011  
APPROVED BY:  
QA/QC APPROVED:

SUBJECT

SCOPE AND APPLICATION

This procedure is designed for the preparation of aqueous samples and TCLP extracts which may contain Extractable Base/Neutral/Acid organic compounds. A measured volume of sample, approximately 1.0 L or 200 mL for TCLP, is serially extracted with Methylene chloride at a pH <2 and again at a pH >11.

MATRIX

Water

REFERENCE

SW-846 Method 3510C  
EPA 625

PRESERVATIVE

Cool to  $\leq 6^{\circ}\text{C}$  but not frozen.

SAMPLE  
COLLECTION

Samples should be collected and stored in one-liter glass containers.

HOLDING TIME

Samples must be extracted within 7 days of collection. Extracts must be analyzed within 40 days following extraction.

TCLP:

From collection to TCLP extraction - 14 days  
From TCLP extraction to preparative extraction - 7 days  
From preparative extraction to analysis - 40 days

DEFINITIONS

See Appendix A

1. 625 Spiking Solution - for this laboratory, this refers to a spiking solution that contains the full list of compounds. The laboratory refers to this as the long list spiking solution.
2. 8270 Spiking Solution - for this laboratory, this refers to a spiking solution that contains fewer compounds. The laboratory refers to this as the short list spiking solution. This spiking solution must not be used when analyzing samples for DOD, AFCEE, South Carolina or when full list spikes are required.

SAFETY

Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards, and all associated protective wear and spill clean-up procedures PRIOR TO the use of any chemical. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables,

poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents must be handled in ventilated hoods.

#### APPARATUS

Water bath - Organomation S-Evap-KD Solvent Evaporator, or equivalent, heated with concentric ring cover and capable of temperature control ( $\pm 5^{\circ}\text{C}$ ). The bath must be used in a hood.

Separatory Funnel - 2 L with Teflon stopcock

Kuderna-Danish (K-D) apparatus

- A) Concentrator tube (receiver)- 10mL, graduated
- B) Evaporation flask - 500mL, attach to tube with blue clamp
- C) Snyder column - three-ball macro

Snyder column - two-ball micro

Glass Wool

Glass funnels or Teflon Funnels - packed with glass wool and fill with Sodium sulfate; rinsed with Methylene chloride; collect rinsate in an Erlenmeyer flask and then discard in the appropriate waste container.

Erlenmeyer flask

100 mL Volumetric Flask

Boiling chips, Teflon or equivalent

pH indicator paper with pH range 2-14

Glass Graduated cylinders - 1000 mL and 100 mL

1.0 mL pipettor or 1.0 mL gas tight syringe

Glass beaker for micro-Snyder process

Hot plate

Glas-col mechanical shaker

1.0 mL class "A" volumetric flask

#### REAGENTS

All organic reagents used must be of Pesticide quality, inorganic reagents must be Reagent grade. Label all containers and squeeze bottles with reagent ID, lot number, date received, date opened and expiration date. Label prepared standards as described in SOP GEN-006. Follow manufacturer's instruction for reagent expiration and storage. When the Manufacturer fails to provide an expiration date, the expiration date will be 12 months from the date opened or sooner if the reagents show signs of deterioration such as change in color, clumping, separation, etc.

Deionized water

Sodium hydroxide solution, 50%(ACS) - Dissolve 1000 g NaOH into 1000 mL of DI water. Record in log book. Label the container with the lot number, expiration date and concentration.

Sodium sulfate (ACS) - granular, anhydrous, heated to 400 °C for 4 hours in a shallow tray, cooled and stored in a glass containers with Teflon lined screw top lids.

Sulfuric acid solution (1:1) - 500mL H<sub>2</sub>SO<sub>4</sub> (sp. gr. 1.84) added slowly to 500mL reagent water.

Methylene chloride

#### STANDARDS

All standards used must be pure material or from prepared certified solutions. The Certificate of Analysis shall be kept on file. Standards are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. Label all working standards with date prepared, concentration, Standard ID number, expiration date and the initials of individual preparing the standard. Label prepared standards as described in SOP GEN-006.

#### Surrogate Standard Spiking Solution

1. Surrogate standards are added to all samples, including QC.
2. Stock surrogate standard - a purchased standard in Methanol containing the following surrogates at the listed concentration:

<u>50 µg/mL</u>	<u>100 µg/mL</u>
Nitrobenzene-d <sub>5</sub>	Phenol-d <sub>5</sub>
2-fluorobiphenyl	2-Fluorophenol
Terphenyl-d <sub>14</sub>	2,4,6-Tribromophenol

3. Store the spiking solutions at 4°C in amber glass bottles with Teflon-lined caps. The solutions must be replaced when expired (manufacturer's date), or sooner if comparison with quality control check samples indicate a problem.
4. Spiking Solutions (625) - stored at ≤ 6°C in amber glass Teflon-sealed containers until manufacturer's expiration date, or sooner if comparisons with quality control check samples indicate a problem.
5. 625 Spiking Solution - full list of compounds. The spiking solution is purchased at a concentration of 100 µg/mL in Methanol. Spike 1.0 mL of the 625 spiking solution into the LCS/LCSD and MS for full list methods. The spiking solution is purchased at a concentration of 100 µg/mL and is spiked directly into the LCS/LCSD and MS.

**Note:** A MSD is not required for method 625.

Additional spiking solutions may be purchased to include additional analytes requested by the client.

6. Base Neutral Spiking Solution (5000 µg/mL), Store at  $\leq 6^{\circ}\text{C}$  in sealed ampules until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.
7. Acid Spiking Solution (7500 µg/mL Store at  $\leq 6^{\circ}\text{C}$  in sealed ampules until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.
8. 8270 Working Spike Solution (100 µg/mL) - add 2.0 mL of Base, Neutral spiking solution and 1.34 mL of Acid spiking solution to a volumetric flask and bring to a final volume of 100 mL with Acetone.

**NOTE:** This is a short list spiking solution and should not be used when analyzing samples for DOD, AFCEE, and South Carolina or when full list spikes are needed.

#### PROCEDURE

1. Rinse all separatory funnels with methylene chloride three times. Discard solvent rinse into solvent waste container.
2. Label each separatory funnel with the sample ID to be extracted. Complete the laboratory extraction sheet with the sample ID. Fill in the extraction sheet information as each item is completed. An extraction sheet is attached.
3. Measure 1000 mL DI water with a graduated cylinder and pour into the separatory funnels labeled Blank, LCS and LCSD.
4. With each extraction batch, a method blank, LCS, LCSD and MS/MSD are included. 625 extraction batches only require a method blank, LCS/LCSD, and MS. Document on the extraction log if insufficient sample is available for a MS and/or MSD.
5. Use a Sharpie to mark the water meniscus on each sample container. This mark will later determine the initial sample volume. For TCLP BNA extraction, use a graduated cylinder to measure 200 mL of the sample.
6. Shake and mix the sample well. Pour the entire contents of the sample container into a labeled separatory funnel. Check the pH of each sample using a



Pasteur pipette and strip of pH paper. Adjust the pH of the sample to a pH of <2 by using 1:1 Sulfuric acid. This should take  $\approx$ 3 mL for most samples.

7. Add 1.0 mL of the surrogate standard spiking solution to each separatory funnel and mix well. Spike the LCS/LCSD and the MS/MSD with 1.0 mL of the required spiking solution. For samples requiring SIMS analysis, add 100  $\mu$ L of the surrogate standard spiking solution to each separatory funnel and 100  $\mu$ L of 625 spiking solution to the LCS/LCSD and the MS/MSD pairs instead of the 1.0 mL required for routine extraction used for 8270 samples.
8. Use 60 mL of Methylene chloride to rinse the sample bottle and graduated cylinder (after rinsing the bottle, transfer to the graduated cylinder).

Transfer this rinse solvent to the separatory funnel. Shake the funnel and vent. Place the funnel on the shaker and set timer for two minutes to extract. Allow the organic (lower) layer to separate from the water phase for at least 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the technician must employ mechanical techniques to effect a phase separation. Optimum techniques depend upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Filter the Methylene chloride extract directly through a prepared glass funnel and collect in a K-D apparatus, fitted with a 10 mL graduated receiver. Using the Methylene chloride rinse bottle, rinse the funnel with a stream of Methylene chloride and collect in the K-D apparatus.

**Note:** If significant amounts of sediment are in the bottom of the sample container, it may not be possible to rinse with the solvent. Contact project management to determine additional instructions from the client. If the client requests only the liquid phase be analyzed, the liquid-liquid extraction can be used. See SOP EXT-004. If the client request both phases analyzed, separate the phases and proceed. Solids are extracted using SOP EXT-001.

9. Add a second 60 mL volume of Methylene chloride to the separatory funnel and repeat the extraction a second time. Allow the organic layer to separate approximately five minutes and then combine this extract with the extract from step 8. Perform a third extraction in the same manner as the second.
10. Add approximately 7 mL of the Sodium hydroxide solution to each funnel. Check the pH and verify it is >11. Additional Sodium hydroxide solution is added if the pH is not >11. Serially extract three times with 60 mL aliquots of Methylene chloride in the same manner as the acid extraction was performed. Collect

and combine the extracts as above and label as BNA.

11. Using the Methylene chloride rinse bottle rinse the funnel and Sodium sulfate thoroughly with a stream of Methylene chloride and collect the rinse in the KD flask.
12. The initial sample volume is determined by filling the sample container with water to the mark indicating the water meniscus. Pour the entire contents into a 1000 mL graduated cylinder. Record the initial volume on the bench sheet.
13. Add one or two Teflon boiling chips to the K-D apparatus. Rinse the 3 ball Snyder column with methylene chloride. Attach the Snyder column to the evaporation flask. Place the K-D apparatus on the water bath. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10 to 15 minutes. At the proper rate of distillation the balls of the column will chatter but the column will not flood with condensed solvent. When the apparent volume of liquid reaches 5.0 - 6.0 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Remove the Snyder column and evaporation flask.
14. Rinse a two-ball micro-Snyder column with Methylene chloride. Add another clean, boiling chip to the concentrator tube and attach the two-ball micro-Snyder column. Place the receiver apparatus on a hot water bath (water bath below boiling) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches about 0.5 mL, remove the apparatus from the water bath and allow it to drain for at least 10 minutes while cooling. Remove the Snyder column. Adjust the final volume to 1.0mL with Methylene chloride in a 1.0 mL Class "A" volumetric tube. Transfer the extract to a Teflon-sealed crimp cap auto-sampler vial and label as BNA, as appropriate.
15. Enter the prep batch information in the LIMS. Sign the extraction sheet. Deliver a copy of the extraction log and the extracts to the GCMS Semivolatiles laboratory. If an analyst is not available to receive the samples, place in the appropriate refrigerator with the extraction sheet.
16. Extracts are analyzed using SOP GCMSSV-001 or GCMSSV-002.

METHOD

PERFORMANCE

1. The recovery of surrogates is used to monitor unusual matrix effects, sample processing problems, etc. The recovery of matrix spiking compounds, when compared to laboratory control sample (LCS) recoveries, indicates the presence or absence of unusual matrix effects.
2. The performance of each 3500 method will be dictated by the overall performance of the sample preparation in combination with the cleanup method and/or the analytical determinative method.

POLLUTION

PREVENTION

See QAPP Section 13.2

WASTE

MANAGEMENT

See SOP GEN-009

METHOD

MODIFICATIONS

The following are modifications of EPA 625 to correspond with SW-846 3510 guidance

1. The acid extraction is performed first. This is done to improve the recovery of phenols that can decompose if the sample is made basic prior to the acid extraction.
2. The base-neutral and acid extracts are combined prior to concentration.

# BASE NEUTRAL/ACID SAMPLE PREPARATION FORM

EXTRACTION DATE/TIME: \_\_\_\_\_

MATRIX: \_\_\_\_\_ WATER \_\_\_\_\_ SOIL \_\_\_\_\_ OTHER \_\_\_\_\_

LEVEL: \_\_\_\_\_ LOW \_\_\_\_\_ MEDIUM \_\_\_\_\_

BATCH #: \_\_\_\_\_

ELEMENT & GROUP #	GCAL ID	INITIAL VOL/WT mL g	FINAL VOLUME (mL)	BN pH	ACID pH	COMMENTS	METHOD
1							
2							LIQUID LIQUID/3520
3							EXTRACTION
4							
5							SEPARATORY FUNNEL/3510
6							EXTRACTION
7							
8							SONICATOR/3550
9							
10							SOXHLET/3540
11							
12							GPC CLEANUP/3640
13							
14							WASTE DILUTION/3580
15							TCLP EXTRACTION FLUID #1
16							
17							TCLP EXTRACTION FLUID #2
18							
19							
20							MECL2/Acetone No:
21							
22							ACETONE LOT NO:
23							
24							
25							MeCL2 Lot No:
26							
27							Sodium Sulfate Lot No:
28							

COMMENTS: SAMPLE PREPARATION INCLUDES DETERMINATION OF SAMPLE VOLUME/WEIGHT, SOLVENT EXTRACTION AND EVAPORATION OF SOLVENT TO FINAL VOLUME

SURROGATE ID _____	8270 SPIKE ID _____	625 SPIKE ID _____	TECHNICIAN _____
VOLUME _____	VOLUME _____	VOLUME _____	DATE _____
CONCENTRATION _____	CONCENTRATION _____	CONCENTRATION _____	
NaOH _____	ACID _____		
	SPIKE WITNESS _____	SUPERVISOR _____	DATE _____

## APPENDIX A

### GLOSSARY

ACCEPTANCE LIMITS - data quality limits specified by the test method or generated by the laboratory. Specified limits placed on an item, process, or service defined in the requirement document.

ACCURACY - a measure of how close a measured value is to a known true value. Accuracy is assessed by means of reference samples and percent recoveries of spiked samples. The degree of agreement between an observed value and an accepted reference value. Accuracy includes combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

ALIQOT - a discrete, measure, or representative portion of a sample taken for analysis.

ANALYTE - the chemical element or compound an analyst seeks to determine; the chemical element of interest.

ANALYTICAL BATCH - the basic unit for analytical quality control, defined as samples that are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. Samples in each batch should be of similar composition (e.g., groundwater, sludge, ash).

ANALYTICAL SAMPLE - any solution or media introduced into an instrument on which an analysis is performed, excluding instrument calibration, initial calibration verification, initial calibration blank, continuing calibration verification, and continuing calibration blank. The following are all analytical samples: undiluted and diluted samples (EPA and non-EPA), predigestion spike samples, duplicate samples, serial dilution samples, analytical spike samples, post digestion spike samples, interference check samples (ICS), CRDL standard for AA(CRA), CRDL standard for ICP(CRI), laboratory control sample(LCS), preparation blank(PB), and linear range analysis sample(LRS).

ANALYST - the designated individual who performs the "hands on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

AREA UNITS - a term used in gas chromatography that indicates the peak area of a compound exiting a chromatographic column. The size or area of the peak is proportional to the amount of analyte in the sample.

ATOMIC ABSORPTION (AA) - a technique for analyzing metal using an element-specific lamp that emits a characteristic light spectrum. A sample is heated in a flame or graphite furnace and the light beam is passed through it. When the sample absorbs light, an energy loss is detected and is translated into a concentration of metal in the sample. This technique detects one metal at a time.

AUDIT - a systematic check to determine the quality of some function or activity. Two basic types are performance audits and system audits. Perform audits involve a quantitative comparison of the laboratory's results to those of a proficiency sample containing known concentration of analytes. A system audit is a qualitative evaluation that normally consists of an onsite review of a lab's quality assurance system and physical facilities.

BACKGROUND CORRECTION - a technique usually employed relative to metals analysis, which compensates for variable background contribution to the instrument signal in the determination of trace elements.

BATCH - environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A batch is composed of a maximum of twenty environmental samples. A preparation batch is composed of environmental sample of the same matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) and/or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices.

BIAS - the deviation of the mean of replicate analytical measurements from a reference analyte concentration. Relative bias is represented by analytical measurement mean minus the reference analyte concentration and the difference divided by the reference analyte concentration. See accuracy and precision.

4-BROMO-FLUOROBENZENE (BFB) - the compound used to check the tuning of the instrument, and is used as a surrogate compound spiked into every sample for volatile analysis. A compound utilized in EPA gas chromatography/mass spectrometry (GC/MS) volatile methods to establish mass spectral instrument performance.

BIOCHEMICAL OXYGEN DEMAND (BOD) - A measure of the amount of oxygen consumed in the biochemical processes that break down organic matter in water. A larger BOD value indicates a greater degree of organic pollution. A related term, BOD<sub>5</sub>, is the amount of dissolved oxygen consumed in five days.

BIOSEED - the bacterial culture used to inoculate a sample for testing.

BLANK - an artificial sample designed to monitor the introduction of artifacts into the measurement process. For aqueous samples, reagent water is used as a blank matrix. There are several types of blanks, which monitor a variety of processes: 1.) *laboratory blank* is taken through sample preparation and analysis only. It is a test for contamination in sample preparation and analyses. 2.) *storage blank* is stored and analyzed with samples at the laboratory. It is a test for contamination in sample storage as well as sample preparation and analysis. 3.) *trip blank* is shipped to and from the field with the sample containers. It is not opened in the field and, therefore, provides a test for contamination from sample preservation, site conditions, and transport as well as sample storage, preparation, and analysis. It is most commonly used for volatile organics. 4.) *field blank* is opened in the field and tests for contamination from the atmosphere as well as those activities listed under the trip blank.

BNA - base, neutral and acid extractable compounds. The term base, neutral and acid refer to the pH condition of the sample undergoing extraction. Certain compounds extract more efficiently from water under acidic conditions. Such compounds are often referred to as "acid extractables."

CALIBRATE - to determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device, or the correct value for each setting of a control knob. The levels of the applied calibration standard should bracket the range of planned or expect sample measurements.

CALIBRATION - The systematic determination of the relationship of the response of the measurement system to the concentration of the analyte of interest.

Instrument calibration performed before any samples are analyzed is called the initial calibration. Subsequent checks on the instrument calibration performed throughout analysis are called continuing calibration verification.

CALIBRATION BLANK (CB) - a volume of reagent water in the same matrix as the calibration standards but without the analyte.

CALIBRATION CURVE - the graphical relationship between the known values, such as concentrations, of a series of calibration standards and their analytical response.

CALIBRATION METHOD - a defined technical procedure for performing a calibration.

CALIBRATION STANDARD - a certified material used to calibrate an instrument.

CERTIFIED REFERENCE MATERIAL (CRM) - reference material, one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30)

CONTINUING CALIBRATION VERIFICATION (CCV) - used to evaluate the performance of the instrument system with respect to a defined set of method criteria. The CCVs are used to verify instrument performance during analysis. The CCV shall be prepared from the same stock standard solution used to prepare the calibration curve.

CHAIN-OF-CUSTODY-Procedures and associated documents designed to trace the custody of a sample from the point of origin to final disposition, with the intent of legally demonstrating that custody remained intact and the tampering or substitutions were precluded.

CHEMICAL OXYGEN DEMAND (COD) - A measure of the oxygen required to oxidize all compounds in water, both organic and inorganic.

COMBINED STANDARD UNCERTAINTY - The standard uncertainty of the analytical measurement result that is the sum in quadrature (square-root-of-the-sum-of-the-squares) of the component standard uncertainties.

CORRECTIVE ACTION - the action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation to prevent reoccurrence.

COVERAGE FACTOR - the numerical factor used as a multiplier of the combined standard uncertainty to expand the uncertainty corresponding to a specific level of confidence. The Student's *t*-distribution is used for determining the coverage factor.

DEMONSTRATION OF CAPABILITY (DOC) - a procedure used to establish the ability of the analyst to generate acceptable accuracy.

DEIONIZED WATER (DI) - water from which the majority of ions have been removed. Water that is contaminant free and used in the laboratory, the resistivity should be  $\leq 18$  mega ohms.

DISSOLVED METALS -- metallic elements determined on a water sample that has been passed through a 0.45-um filter.

DISSOLVED OXYGEN (DO) - the oxygen freely available in water, an indicator of water quality.

DISSOLVED SOLIDS - disintegrated organic and inorganic material contained in water. Excessive amounts make water unfit to drink or use in industrial processes.



DRY WEIGHT - the weight of a sample based on percent solids. The weight of a sample after drying in an oven at a specified temperature.

DUPLICATE MEASUREMENT - a duplicate measurement is a second measurement made on the same or identical sample of material to assist in the evaluation of measurement of variance.

DUPLICATE SAMPLE - two samples taken from the same population and carried through certain stages of sampling and testing. Duplicate sample include field co-located duplicate samples, field-split duplicate samples, and laboratory duplicate subsamples.

EXPANDED UNCERTAINTY - the quantity defining an interval enveloping the analytical measurement that captures a large fraction of the distribution of analyte concentrations that could be attributable to the quantity measured. The combined standard uncertainty is multiplied by the coverage factor to calculate the expanded uncertainty.

EXTERNAL STANDARDS - a method of quantifying chromatographic data in which standards of known concentration are analyzed prior to unknown samples. The chromatographic peak area (or height) of a sample component is compared to a calibration curve of a peak area constructed from the standard data for that component. This comparison allows the concentration of the component in the sample to be determined.

FECAL COLIFORM BACTERIA - bacteria found in mammals' intestinal tracts. Their presence in water or sludge is an indicator of pollution and possible contamination by pathogens.

FLAME IONIZATION DETECTOR (FID) - a gas chromatography detector in which the column effluent gas is mixed with hydrogen and burned in air or oxygen. The ions and electrons produced in the flame generate an electric current proportional to the amount of materials in the detector. The FID responds to nearly all organic compounds, but it does not respond to air and water, which makes it exceptionally suited to environmental analysis.

FLASHPOINT - the lowest temperature at which a flammable liquid gives off sufficient vapor to form an ignitable mixture with air near its surface or within a vessel. Combustion does not continue.

GAS CHROMATOGRAPHY (GC) - a technique for detecting organic compounds by using their physical and chemical properties to separate a mixture. The compounds are identified and quantified with various types of detectors as they exit the chromatograph. Selection of detectors is dependent on the particular compounds of interest.

GAS CHROMATOGRAPHY MASS SPECTROMETRY (GCMS) - a technique in which sample analytes are bombarded with electrons as they exit a gas chromatography column and are fragmented into characteristic ion patterns. The mass spectrometer is the detector. It can determine which fragments are present and therefore the identity of the compounds.

GLUCOSE GLUTAMIC ACID (GGA) - used as a laboratory control standard in BOD procedures.

GRAPHITE FURNACE - a technique used for the analysis of metals. An atomic absorption spectrophotometer heats the sample within a graphite tube using an electrical current. It is also commonly called a flameless furnace and

generally provides greater sensitivity for certain metals than flame or inductively coupled argon plasma techniques.

GRAVIMETRIC - analyses based on the direct or indirect weighing of the analyte in question. This technique usually requires the use of an analytical balance with a sensitivity of 0.1 mg or better.

HALL ELECTROLYTIC CONDUCTIVITY DETECTOR - an element-selective gas chromatography detector primarily intended for trace analysis of organic compounds containing chlorine, nitrogen or sulfur. In operation, this detector pyrolyzes the column effluent gas into soluble electrolytes that are dissolved in a stream of deionized liquid. The observed change in electrical conductivity, proportional to the amount of material present, is measured.

HAZARDOUS WASTE - waste regulated under RCRA that can pose a substantial or potential hazard to human health or the environment when improperly managed. Such wastes possess at least one of four characteristics (ignitability, corrosivity, reactivity, or toxicity) or appear on special EPA hazardous waste lists. The term is not interchangeable with hazardous substances or material.

HEADSPACE - Any area in a container not completely filled by the sample in which gases can collect.

HEAVY METALS - metallic elements with high atomic weights, i.e., mercury, chromium, cadmium, arsenic, and lead. They can damage the health of plants and animals at low concentrations and tend to accumulate in the food chain.

HOLDING TIME - the maximum times that samples may be held before analysis and still be considered valid or not compromised. The storage time allowed between sample collection and sample analysis when the designated preservation and storage techniques are employed.

HYDROCARBONS - chemical compounds that consist entirely of carbon and hydrogen.

ICP - Inductively coupled plasma is a type of instrument used for metals analysis. Because the temperature of the plasma is considerable higher (10,000°K) than the temperature of a flame atomic absorption spectrophotometer, it is especially useful for refractory metals. Some instruments are also capable of performing simultaneous multielement analysis.

ICP-MS - Inductively coupled plasma mass spectrometry (ICP-MS) is a type of mass spectrometry highly sensitive and capable of the determination of a range of metals and several non-metals at concentrations below one part in 10<sup>12</sup> (part per trillion). It is based on coupling together an inductively coupled plasma as a method of producing ions with a mass spectrometer as a method of separating and detecting the ions. In trace elemental analysis, the method has advantages of high speed, precision and sensitivity.

IGNITABLE - capable of burning or causing a fire.

INORGANIC CHEMICALS - chemical substances of mineral origin, unlike organic chemicals whose structure relies on carbon atoms.

INITIAL CALIBRATION VERIFICATION (ICV) - also referred to as the Independent Calibration Verification for this laboratory. A standard solution used to verify the calibration curve derived from a source independent of the instrument calibration standard. The ICV is used to quantify second source standard variance and bias.

INSTRUMENT CALIBRATION STANDARD - a reference material used to standardize an analytical instrument.

INSTRUMENT DETECTION LIMIT (IDL) - a term utilized in the EPA Inorganic Contract Laboratory Program. The IDL is three times the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration of three to five times that of the IDL on three nonconsecutive days with seven consecutive measurements per day. The IDL is the concentration equivalent to a signal, due to the analyte of interest, which is the smallest signal that can be distinguished from background noise by a particular instrument. The IDL should always be below the method detection limit, and is not used for compliance data reporting, but may be used for statistical data analysis and comparing the attributes of different instruments.

INSTRUMENT PERFORMANCE CHECK - The analyses of one of the ICSs to verified initial and continuing calibration (CCV). The IPC is used to quantify the instrumental testing repeatability variance and bias.

INSTRUMENT TUNING - a technique used in gas chromatography/mass spectrometry procedures to verify that the instrument is properly calibrated to produce reliable mass spectral information. See 4-Bromofluorobenzene under "B".

INTERNAL STANDARDS - compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for volatile organics), and sample extract (for semivolatiles) at a known concentration, prior to analysis. Internal standards are used as the basis for quantification of the target compound.

IONIZATION - Utilized in mass spectrometry to fragment analyte molecules into smaller segments. These smaller mass segments are then separated and plotted to form a "mass spectrum" which is used to identify the parent molecule. Electron impact is one example of ionization used in mass spectrometry. In more technical terms. Ionization is the process by which neutral atoms or groups of atoms become electrically charged, either positively or negatively, by the loss or gain of electrons.

ISOMERS - chemical compounds with the same molecular weight and atomic composition by differing molecular structure, e.g., n-pentane and 2-methylbutane.

LABORATORY CONTROL SAMPLE (LCS) - a clean-matrix reference sample spiked with a standard with an established analyte concentration. The LCS is carried through the entire chemical preparation and testing procedures. The LCS is used to quantify the variance and bias of the chemical preparation and instrumental testing stages without matrix interference. Also called a Laboratory fortified blank (LFB).

LABORATORY CONTROL SAMPLE DUPLICATE (LCSD) - a duplicate of the laboratory control sample in a clean matrix that is carried through the entire chemical preparation and testing procedures.

LABORATORY DUPLICATE SAMPLE (LAB DUPLICATE) - a portion of the collected sample that is carried through the chemical preparation and testing. The laboratory duplicate subsample is used to quantify the variance of the chemical preparation and instrumental testing stages with matrix interferences.

LEACHATE - a liquid that results from water collecting contaminants as it trickles through wastes, agricultural pesticides or fertilizers. Leaching may occur in farming areas, feedlots, and landfills, and may result in hazardous substances entering surface water, groundwater or soil.

LDEQ - Louisiana Department of Environmental Quality

LIBRARY SEARCH - a technique in which an unknown mass spectrum of a compound is compared to the mass spectra of compounds contained in a computer library in an

effort to identify the compound. Compounds identified in this manner are referred to as tentatively identified compounds (TICs).

LIMIT OF DETECTION (LOD) - an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent.

LIMIT OF QUANTIFICATION (LOQ) - the minimum levels, concentrations, or quantities of a target variable (e.g. target analyte) that can be reported with a specified degree of confidence..

LIMS - laboratory information management system. Horizons is the LIMS used by GCAL.

LINEAR CALIBRATION RANGE - the concentration range over which the instrument response is linear.

LOG-IN - the receipt and initial management of an environmental sample. It generally includes identifying who sent the sample; maintaining chain-of-custody; checking report and invoice information; recording analysis requested, including methodology and special instructions; and assigning a discreet in-lab identification, usually a number or bar code.

MASS SPECTRUM - a plot of ion mass/charge ratio versus intensity. A fragmentation pattern results from the particles whose mass distribution is characteristic of the parent molecule. Qualitative information is provided by a mass spectrum.

MATERIAL SAFETY DATA SHEET (MSDS) - a compilation of information required under the OSHA Communication Standard on the identity of hazardous chemicals and their associated health and physical hazards, exposure limits and precautions.

MATRIX - The physical characteristics or state of a sample - e.g., water, soil, sludge.

MATRIX INTERFERENCE - the influence of the sample matrix or sample components upon the ability of qualitatively identifies and quantitatively measures compounds in environmental samples.

MATRIX MODIFIERS - chemicals added to samples for metals analysis, which are used to lessen the effects of chemical interference, viscosity and surface tension.

MATRIX SPIKE (MS) - aliquot of a sample fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery of the spike. Matrix spiked samples are used to quantify the variance and bias of the chemical preparation and testing stages with matrix interference.

MATRIX SPIKE DUPLICATE (MSD) - a second aliquot of the same matrix as the matrix spike that is spiked to determine the precision of the method.

METHOD DETECTION LIMIT (MDL) - the minimum concentration of compound that can be measured and reported within 99% confidence that the value is above zero and is determined from analysis of a sample in a given matrix containing the analyte.

MUST - denotes a mandatory requirement.

NARRATIVE - in an analytical report, a descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

NUTRIENT - any substance assimilated by living things that promote growth. The term is generally applied to nitrogen and phosphorus in wastewater, but is also applied to other essential and trace elements.

ORGANIC - generally, any compound that contains carbon bonded to a hydrogen or halogen atom.

OXIDATION - the process in chemistry whereby electrons are removed from a molecule.

PCBs - Polychlorinated biphenyl, a group of toxic persistent chemicals used in transformers and capacitors for insulating purposes and in gas pipeline systems as a lubricant. Sale of PCBs for new uses was banned by law in 1979.

PERCENT RECOVERY - a measure of accuracy that is calculated as the measured value relative to the true value expressed as a percent.

PERFORMANCE AUDIT - a quantitative evaluation of a measurement system that involves the analysis of standard reference samples or materials which are certified as to their chemical composition or physical characteristics.

PROFICIENCY TEST (PT) SAMPLE - a sample of known composition (unknown to the laboratory), provided by an external source, which is used to evaluate lab performance. For certification purposes the PT sample is purchased from an approved provider.

pH - a numerical designation of relative acidity and alkalinity. A pH of 7.0 indicates precise neutrality. Progressively higher values indicate increasing alkalinity and lower values increasing acidity.

POLLUTANT - generally, any substance introduced into the environment that adversely affects the usefulness of a resource.

PRACTICAL QUANTITATION LIMIT (PQL) - the lowest level that can be reliably achieved within specified limits of precision and accuracy routine laboratory operating conditions. The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value.

PRECISION - a measure of the ability to reproduce analytical results. It is generally determined through the analysis of duplicate samples. The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range in either absolute or relative terms.

PRESERVATIVE - a chemical or reagent added to a sample to prevent or slow decomposition or degradation of a target analyte or a physical process. Physical and chemical preservation may be used in tandem to prevent sample deterioration.

PURGE AND TRAP - a technique used in the analysis of volatile organic where analytes are purged from a sample by means of an inert gas and trapped on a sorbent column. The sorbent is then flashheated and the analytes are transferred onto a gas chromatographic column for separation and identification.

PURGEABLE ORGANIC - an organic compound that is generally less than 20% soluble in water and has a boiling point at or below 200°C. A volatile organic. An organic compound is generally considered to be purgeable if it can be removed from water using the purging process.

QC BASED NESTED APPROACH - a procedure for estimating the uncertainty of measurement using quality control data. The procedure is described in SOP QA-013.

QUALITY ASSURANCE (QA) - all those planned and systematic actions necessary to provide adequate confidence in laboratory results. An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets the defined standards of quality with a stated level of confidence.

QUALITY ASSURANCE PROGRAM PLAN - a written assembly of management policies, objectives, principle and general procedures that outlines how the laboratory intends to generate data of known and accepted quality.

QUALITY CONTROL (QC) - the overall system of technical activities whose purpose to measure and control the quality of a product or service so that it meets the needs of the users.

QUALITY CONTROL CHART - a graph of analytical measurement results for a specific QC standard plotted sequentially with upper and lower control limits ( $\pm 3SD$ ). A central line that is the best estimate of the average variable plotted, and upper and lower marginal exceedence limits ( $\pm 4SD$ ) are usually included in the Quality Control Chart. The chart may also include upper and lower warning limits ( $\pm 2SD$ ).

QUALITY SYSTEM - a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items) and services. The quality system provides the framework for planning, implement and assessing work performed by the organization and for carrying out required QA and QC.

$r^2$  - Correlation Coefficient Squared - parameter used to evaluate the fit of linear or quadratic regression curve fit of initial calibration data.

RAW DATA - any original information from a measurement activity or study recorded in a laboratory note book, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfilm copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

REACTIVITY - the tendency of a chemical to explode under normal management conditions, to react violently when mixed with water, or to generate toxic gases.

REAGENT WATER - water in which an interference is not observed at or above the minimum quantitation limit of interest.

REFERENCE MATERIAL - a traceable standard with an established analyte concentration. Material or substance whose property values are sufficiently homogenous, stable and well established to be used for the calibration of an apparatus, the assessment of a measurement method or for assigning values to materials (ISO Guide 30)

REFERENCE METHOD - a method of known and documented accuracy and precision issued by an organization recognized as competent to do so.

RELATIVE RETENTION TIME - a measure of the shift in retention time of an analyte when referenced to an internal standard.

RELATIVE RESPONSE FACTOR (RRF) - a measure of the relative mass spectral response of an analyte compared to its internal standard. RRFs are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

REPLICATE ANALYSES - two or more results representing the same sample parameter. Replicate analyses are used to quantify the analytical measurement repeatability precision.

RPD - Relative Percent Difference, a relative difference between two analyses calculated by dividing the absolute difference of those two results by the average of their two values, then multiplying by 100.

RESOLUTION - the degree of separation between peaks eluting from a chromatographic column. Sufficient resolution between peaks is required for proper quantitation of unknown analytes.

RETENTION TIME - a term used in gas and liquid chromatography describing the time elapsed from the sample injection until the specific compound elutes or exits the chromatographic column at the detector. Each compound has a characteristic retention time on a specific column; therefore, this information is used to qualitatively identify the compounds in the sample.

SAMPLE - portion of material collected for chemical analysis, identified by a unique number assigned by the LIMS.

SHALL - denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled.

SHOULD - denotes a guideline or recommendation whenever noncompliance with the specification is permissible.

SOLID WASTE - non-liquid, non-soluble materials, ranging from municipal garbage to industrial waste, that contain complex, and sometimes hazardous, substances. Solid wastes include sewage sludge, agricultural refuse, demolition wastes, mining residues, and even liquids and gases in containers.

SOLVENT - a substance, usually liquid, capable of dissolving or dispersing one or more other substances.

SPIKE - a known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

STANDARD CURVE - a curve that plots concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by diluting the stock analyte solution in graduated amounts that cover the expected range of the samples being analyzed. The calibration standards must be prepared by using the same type of acid or solvent at the same concentration as for the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

STANDARD OPERATING PROCEDURE - a detailed written description of how a laboratory executes a particular procedure or method intended to standardize its performance.

STANDARDIZED REFERENCE MATERIAL (SRM) - a certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method.



STANDARD UNCERTAINTY - the analytical measurement uncertainty expressed as a standard deviation. The relative standard deviation represents the relative standard uncertainty.

SURROGATE - an organic compound similar to the analyte of interest in chemical composition, extraction and chromatography, but not normally found in environmental samples. Primarily used in chromatography techniques, the surrogate is spiked into quality control blanks, calibration and check standards, samples (including duplicated and QC reference samples) and spiked samples before analysis. A percent recovery is calculated for each surrogate.

SUSPENDED SOLIDS - small pollutant particles that float on the surface of, or are suspended in, sewage or other liquids. They resist removal by conventional means.

TARGET COMPOUND - specific compounds that are to be quantified in a sample based on a standard list of potential compounds.

TENTATIVELY IDENTIFIED COMPOUNDS (TICs) - compounds detected in samples that are not target compounds, internal standards, system monitoring compounds or surrogates. TICs usually consist of up to 30 peaks that are greater than 10% of the peak areas, or heights, of the nearest internal standard. They are subjected to mass spectral library searches for tentative identification. A client may specify the number of unknown peaks in its samples it wishes the laboratory to tentatively identify.

TOTAL METALS - metallic elements that have been digested prior to analysis.

TYPE A EVALUATION UNCERTAINTY - the method of evaluation of uncertainty by the statistical analysis of a series of test results.

TYPE B EVALUATION OF UNCERTAINTY - the method of evaluation of uncertainty by means other than statistical analysis.

UNCERTAINTY - the parameter associated with the analytical measurement results that characterizes the dispersion of the values that could be reasonable attributed to the quantity measured.

UNCERTAINTY INTERVAL - the range of analyte concentrations that an analytical measurement could represent at a specified level of confidence. The relative standard deviation is used to represent the relative standard uncertainty in the QC-based Nested Approach.

VALIDATION - the process for evaluating the completeness, correctness, and conformance/compliance of a specific data set against the method, procedural, or contractual specifications. It essentially evaluates performance against predetermined specifications.

VOA - volatile organic analysis

VOA BOTTLE - a vial used to contain samples for volatile organic analysis.

VOLATILE COMPOUNDS - compounds amenable to analysis by purge and trap. Synonymous with purgeable compounds.

VOLATILE ORGANIC COMPOUND (VOC) - any organic compound that participates in atmospheric photochemical reactions, except for those designated by the EPA Administrator as having negligible photochemical reactivity.

WET CHEMISTRY - procedures that involve distillations, colorimetric determinations and titrimetric measurements. Examples are chloride, nitrates, sulfates and biochemical oxygen demand.



STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF SEMI-VOLATILE MASS SPEC SAMPLES FOR 8270D	REVISION NO. 7
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GULF COAST ANALYTICAL LABORATORIES, INC  
GCMS SEMI-VOLATILES  
STANDARD OPERATING PROCEDURE

PROCEDURE: GCMSSV-004  
PAGE: 1 OF 17  
EFFECTIVE DATE: 2/13/12  
APPROVED BY:  
QA/QC APPROVED:

SUBJECT

SCOPE AND APPLICATION

EPA Method 8270D (SW-846) is used to quantitatively analyze most neutral, acidic and basic (BNA) organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, ethers, aldehydes, ketones, anilines, pyridines, quinolines, aromatic nitro compounds and phenols, including nitrophenols. BNAs are generally extracted using SOPs EXT-001 for solids and EXT-003 for aqueous samples. High-level wastes are prepared by waste dilution. A list of target analytes is available in the LIMS.

Theory & Principle - Gas chromatographic analysis utilizing Mass Selective Detectors eliminates, to a great extent, the secondary column and detector for confirmation. Mass selective detectors ionize each compound to produce its own unique mass fragmentation profile. Direct injection by autosampler is the introduction method for samples to be analyzed via EPA Method 8270 (SW-846). The polarity of the column phase, molecular weight and temperature determine the elution order. The end of the column is positioned so that the eluting compounds are ionized immediately. The ensuing charged ions are directed along an electronically charged quadrupole to the electron multiplier, where the signal is amplified. The MSD can be used in selective ion monitoring and scan mode. In the scan mode, the detector scans for all ions in a selected range. The resulting ion fragmentation profiles are compound fingerprints. The selective ion monitoring (SIM) mode allows for the detection of two to three selected ions per compound in each retention window resulting in lower detection limits. Isomeric compounds must also depend upon retention times as a determining factor. Semi-volatile analysis primarily involves medium to high molecular weight compounds that are readily soluble in methylene chloride. The BNA sample preparation process utilizes the very polar methylene chloride, to extract the target compounds. Helium is used as the carrier gas.

MATRIX

Water and Solid Extracts

STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF SEMI-VOLATILE MASS SPEC SAMPLES FOR 8270D	REVISION NO. 7
REFERENCE	SW846 8270D and 8000B
HOLDING TIME & PRESERVATIVE	<p>Waters - store at 0°C to 6°C for up to but not exceeding seven days before extraction. Hold up to 40 days after extraction at -15 ± 5°C in sealed vials. Protect from light.</p> <p>Solids - store at 0°C to 6°C for up to but not exceeding fourteen days before extraction. Hold up to 40 days after extraction at -15 ± 5°C in sealed vials. Protect from light.</p>
DEFINITIONS	See Appendix A for Glossary.
ANALYTE LIST	See Appendix IV.
SAFETY	<p>Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards, and all associated protective wear and spill clean-up procedures PRIOR TO the use of any chemical. In all cases consult the applicable material safety data sheet (MSDS) and the supervisor or safety officer. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures, solvents and chemicals must be handled in ventilated hoods.</p>
INTERFERENCES	<p>Interferences due to contamination are monitored by analysis of a method blank performed with each batch. Interferences with the parent (quantitation) ion may occur with co-elution and must be monitored by the analyst.</p>
INSTRUMENTATION & APPARATUS	<p>Agilent/5973-6890N or 5975-6890N GC/MS with autosampler  Target data acquisition system  DB-5MS or RTX Ssil MS columns  Volumetric flasks  Syringes - gas-tight  Pasteur pipets  Disposable glass micropipettes  2 mL autosampler vials with crimp tops  Bottles - amber glass with PTFE-lined screw tops or crimp tops</p>
REAGENTS	<p>All organic solvent shall be of pesticide grade or equivalent. Label all containers and squeeze bottles with reagent ID, lot, and expiration date.</p>

STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF SEMI-VOLATILE MASS SPEC SAMPLES FOR 8270D	REVISION NO. 7
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Methylene chloride (DCM)  
Helium gas

#### STANDARDS

All standards used must pure material or from prepared certified solutions. The certificate of analysis shall be kept on file. Follow manufacturer's instruction for standard expiration and storage. Label all working standards using completed standard labels.

1. All stock standards are received in glass sealed ampoules.
  - A. The following stock standards are used to prepare solutions for calibration and CCV samples:
    - A.1 Stock calibration standard -A custom-made stock standard (TCL-200 ug/ml level) is purchased from NSI. It is stored in an amber vial in the freezer at  $-15^{\circ} \pm 5^{\circ}\text{C}$ , until the documented manufacturer's expiration date. This standard includes the surrogates.
    - A.2 Stock calibration standards - 2000 ug/mL -for Appendix IX compounds - commercially prepared stored at  $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$  until the documented manufacturer's expiration date.
    - A.3 Additional standards may be run that are project specific. All standards shall be certified and stored according to the manufacturer's instructions.
  - B. Independent stock standards - standard mixes identical to those listed above from a secondary source or a different lot number from the same manufacturer. These standards are used to prepare the ICV.
  - C. Stock Internal Standard Mixture: 4000 ug/mL - commercially prepared in methylene chloride, stored at  $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$  until the documented manufacturer's expiration date.
  - D. Tuning Standard: 1000 ug/mL - commercially prepared in methylene chloride, stored at  $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$  until the documented manufacturer's expiration date.
2. Working standards - working standards are prepared by diluting the stock standard as described below.
  - A. Assemble the necessary glassware, syringes and solvent. Spectroscopic grade methylene chloride is used in making all standards.
  - B. Ensure that all glassware and syringes are clean and free of moisture by rinsing with at least three aliquots of methylene chloride. Volumetric glassware must never be stored in or placed in heated ovens. The use of dedicated glass will help prevent cross-contamination of standards.

- C. Fill the volumetric glassware to approximately 75 percent volume.
  - D. Add the proper volume of standards using a syringe.
  - E. After the addition of standard, dilute to the calibration mark in the volumetric flask
  - F. Insert the glass stopper and mix.
  - G. Using a Pasteur pipette, transfer the standard to a 2 mL ampoule. Seal the ampoule immediately.
  - H. Label the vial with the following information:  
Expiration Date  
Standard ID #
  - I. Store the vial the same as the stock standard.
  - J. Dispose of excess materials in accordance with established laboratory procedures.
  - K. Always quantitate new standards against known standards to ensure accurate concentration levels.
  - L. Fill out the standards logbook in accordance with proper laboratory procedure and enter required information in LIMS.
3. The working standards are prepared in the following manner:
- A. Working Appendix IX standards - prepare a 200 ug/mL standard mix from the 2000 ug/mL stock standards.
  - B. Working standards from 0.2 ug/mL to 160 ug/mL are prepared as needed before a calibration is performed. They are stored in amber vials in the freezer at  $-15^{\circ} \pm 5^{\circ}\text{C}$ . To make 1.0 mL of each "working standard" of a calibration, do the following:
    - Add 0.001 mL of TCL-200 to 0.999 mL DCM\* for a 0.2 ug/mL standard
    - Add 0.005 mL of TCL-200 to 0.995 mL DCM\* for a 1 ug/mL standard
    - Add 0.05 mL of TCL-200 to 0.95 mL DCM\* for a 10 ug/mL standard
    - Add 0.25 mL of TCL-200 to 0.75 mL DCM\* for a 50 ug/mL standard
    - Add 0.40 mL of TCL-200 to 0.60 mL DCM\* for an 80 ug/mL standard
    - Add 0.60 mL of TCL-200 to 0.40 mL DCM\* for a 120 ug/mL standard

- Add 0.80 mL of TCL-200 to 0.20 mL DCM\* for a 160 ug/mL standard
- The high concentration standard is the stock standard - 200 ug/mL
- Add 10 µL of 4000 ng/µL Internal Standard Mix to each 1.0mL of the "working standard" before shooting

To make the calibration standards for SIM analysis, the TCL standard is used to make the following working and calibration standards. **Please note that for South Carolina samples, SIM analysis is not allowed.**

- Add 125 µL of TCL-200 to 4.875 mL DCM\* for a 5 µg/mL standard
- Add 200 µL of the 5 µg/mL standard to 800 µL DCM for a 1 µg/mL standard.
- Add 100 µL of the 5 µg/mL standard to 900 µL DCM for a 0.5 µg/mL standard.
- Add 20 µL of the 5 µg/mL standard to 980 µL DCM for a 0.1 µg/mL standard.
- Add 10 µL of the 5 µg/mL standard to 990 µL DCM for a 0.05 µg/mL standard.
- Add 10 µL of 4000 µg/mL Internal Standard Mix to each 1.0mL of the "working standard" before shooting

C. The Appendix IX calibration standards are prepared in the same manner from the 200ug/mL working standard. The 200 ug/mL standard will serve as the high concentration standard.

D. Additional stock standards shall be purchased for compounds not included in the mixes.

#### GC RECOMMENDED CONDITIONS

The following lists conditions that may be used in the lab. Conditions vary for each instrument. Adjustments are made when new columns are installed. The analyst is allowed to modify settings to optimize operating conditions.

Initial Temperature: 40°C

Rate A: ~40°C/min to ~245°C

Hold: ~4 to 5 minutes

Injector Temperature: ~ 250°C

Injection volume: 1 uL

#### PROCEDURE

##### Analysis Procedure

1. Injection port maintenance is performed every day of use at a minimum. This includes replacement of septa and liner.
2. Tuning - Before analysis begins each GC/MS must meet tuning criteria specified in the method. A solution containing 50



ug/ml each of Decafluorotriphenylphosphine (DFTPP), 4,4'-DDT, Pentachlorophenol and Benzidine is analyzed. The DFTPP tuning criteria listed below must be met before proceeding with analysis. DDT breakdown shall not exceed 20% and is calculated as:

$$\% \text{Breakdown} = \frac{\text{area DDE} + \text{area DDD}}{\text{area DDE} + \text{area DDD} + \text{area DDT}} \times 100$$

Evaluate the tailing factor for Benzidine and Pentachlorophenol. Check that response is normal and that there is not excessive tailing. If tailing is observed, calculate tailing factors using the equation below. The calculated tailing factor for Benzidine and Pentachlorophenol shall be less than 2. Excess tailing indicates the need for instrument maintenance. If there is no visible tailing document this.

$$\text{Tailing Factor} = \frac{BC}{AB}$$

Analyze The DFTPP result using three scans, the apex and the scan prior and following the apex and subtracting the background no more than 20 scans prior to elution of the DFTPP peak. If the DFTPP fails the criteria listed below, then the analyst shall re-tune the instrument, then run another DFTPP. If the DFTPP still fails the criteria, the analyst shall perform instrument maintenance. These criteria must be met once for every 12 hours of analysis time.

#### DFTPP KEY ION ABUNDANCE CRITERIA

<u>MASS</u>	<u>ION ABUNDANCE CRITERIA</u>
51	30 to 60% of mass 198
68	less than 2% of mass 69
69	mass 69 relative abundance
70	less than 2% of mass 69
127	40 to 60% of mass 198
197	less than 1% of mass 198
198	base peak, 100% relative abundance
199	5 to 9% of mass 198
275	10 to 30% of mass 198
365	greater than 1% of mass 198
441	present but less than mass 443
442	greater than 40% of mass 198
443	17 to 23% of mass 442

3. Calibration - The initial calibration consists of a series of standards analyzed at the concentration noted in the Standards Section 3.B-D. An initial calibration curve for each target

analyte must be analyzed and evaluated before any peak for that analyte can be quantitated.

The calibration range is defined as the on-column concentration range adjusted for sample prep. Because nine calibration standards are used, the analyst has the option of eliminating several of the points using the criteria below.

- a) Points may not be removed from the middle of the curve.
- b) Calibration acceptance criteria must be met as described below.
- c) The lowest calibration point must support the lowest reporting limit needed in the associated samples.
- d) The QC spike amount must be within the calibration range.

Replacing points in the middle of a curve is not allowed unless the analyst can document a technical issue at the time of analysis or spiking of the standard. The new point must be analyzed in the same analytical batch. If the problem appears to be associated with a single standard, that one standard may be reanalyzed. Replacing the standard may be necessary in some cases.

Prepare the calibration standards and Add 10 µL of 4000 ug/ml Internal Standard Mix to each.

When performing analysis in the SIM mode, use the same retention windows used in scan analysis and at least two of the ions listed in Table 1 of SW-846 8270D. The primary ion is used for quantitation and the secondary ion(s) as confirmation.

The calibration curve is now ready for analysis.

The acceptance criteria for initial calibration must be satisfied before analysis of samples begin. Select projects may have additional or more stringent criteria that must be achieved for the applicable samples. See SOP GEN-019.

A. Initial Calibration Acceptance Criteria:

A.1 Minimum target compound response factor requirements are shown on Appendix 1.

B. Additionally one of the following options must be met.

Always attempt to meet calibration criteria using the average response factor. If the average response factor does not pass, options B.2 maybe evaluated. The calibration options and requirements are as follows:

B.1 Average Response Factor Calibration. For each of the standards, calculate the response factor of each compound. Calculate the average of a minimum of five response factors and the standard deviation across the selected five response factors. Use the average RF and the standard deviation to calculate the percent

relative standard deviation (%RSD). All equations can be found in the Calculation section. When the five (or more) response factors of the standards demonstrate less than 20% RSD for a target analytes, linearity through the origin can be assumed. If the RSD for any analyte is greater than 20%, the analyst may wish to review the results for those analytes to ensure that the problem is not associated with just one of the initial calibration standards.

- B.2 For those compounds that the RSD exceeds 20%, a linear regression equation that is not forced through the origin may be used. The correlation coefficient must be at least 0.990 for the curve to be acceptable. When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve. It is not necessary to re-analyze a low concentration standard, rather the data system can recalculate the concentrations as if it were an unknown sample. The recalculated concentration of the low calibration point should be within  $\pm 30\%$  of the standard's true concentration. Analytes which do meet the minimum quantitation calibration re-fitting criteria should be considered "out of control" and corrective action such as redefining the lower limit of quantitation and/or reporting those "out of control" target analytes as estimated when the concentration is at or near the lowest calibration point may be appropriate.

NOTE: If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient of 0.990; then the chromatographic system is considered too reactive for analysis to begin. System maintenance should be performed and the calibration procedure repeated.

- B.3 A quadratic curve fit may be used if the correlation coefficient is at least 0.990. A minimum of a six-point calibration is used if this option is chosen and the curve shall not be forced through zero. As part of the evaluation, check the y-intercept (b) and any negative factors (b or M). Negative factors may result in false negatives and positives. Large shifts in the y-intercept from zero will impact the data. If the intercept is greater than half the reporting limit, this option shall not be used. Check the required report limit any impacts on the data. False positives shall be indicated in the case narrative. Please note that quadratic curves may not be employed for an analyte if normally it passes calibration criteria for

average response or linear regression. For South Carolina samples, quadratic curve fits are not allowed.

- C. If the calibration criteria are not met, possible corrective action includes cleaning the injection port, cutting off the first 4 inches of column or source cleaning, and adjusting the calibration range.
- D. Check the position of the retention time by using the mid-point of an initial calibration. Check and reset as necessary after source cleaning or column maintenance.

#### 4. INITIAL CALIBRATION VERIFICATION

Immediately following the initial calibration procedure or before sample analysis, the analyst shall perform initial calibration verification (ICV). This will consist of a solution containing all target analytes prepared from a standard that is independent from the initial calibration.

The ICV must be analyzed following the initial calibration. The ICV recovery must be 70-130%.

If any target analyte recovery is outside the control limits, corrective action must be taken. This may include instrument maintenance, re-analysis of the ICV or initial calibration, or re-preparation of the standards involved. If holding time or agreed project due dates will not be met because of ICV failure, the client must be contacted and approve of proceeding with the analysis. Note all failures in the case narrative.

#### 5. CONTINUING CALIBRATION VERIFICATION -

If the instrument has been previously calibrated, then a continuing calibration is required. A mid-level standard, 50 ug/mL for scan and 0.5 ug/mL for SIM is analyzed and checked against the initial calibration for verification every 12 hours. Target compounds must have a %D of  $\leq 20$  and must have a minimum RF as listed in Appendix 1. If there are any deviations, the calibration check standard must be re-analyzed or if necessary, column maintenance should be performed. If the calibration check standard still fails the criteria, then another initial calibration must be run. If there are no hits in the sample and the target compound is above 20% D, the sample may be reported with a narrative.

The retention time for any internal standard shall not vary any more than 30 seconds from the last calibration check (12 hours). The extracted Ion Current Profile area for any internal standard must not deviate by more than a factor of two.

## 6. SAMPLE ANALYSIS

Samples are analyzed after an acceptable CCV. Add 10 uL of the internal standard mixture to each extract before analysis.

Compound identification - positive hits are achieved by the following criteria:

- A. The quantitation ion used shall be the ion listed in the reference method unless there are interferences. Qualifier ions shall be used as a replacement, if possible. If the quantitation ion used is not the ion listed in the reference method, this shall be documented.
- B. The characteristic m/z's must maximize within one scan of each other.
- C. The retention time of the compound in the sample must be within  $\pm 0.50$  minutes of the retention time of that compound in the standard and have an RRT of  $\pm 0.06$ .
- D. The relative abundance of the three characteristic m/z's in the sample must fall within  $\pm 30\%$  of the relative abundances of that parameter in the standard.
- E. Structural isomers can be differentiated only if the height between the baseline and the valley between the pairs is less than 50% of the average of the two peak heights. Otherwise they must be reported as isomeric pairs.
- F. If samples have compounds that are above the calibration range, the sample must be diluted. A portion of the sample, measured with a syringe, is diluted to one mL with DCM. Additional internal standard is added to maintain a concentration of 40 $\mu$ g/L. Surrogate recovery is affected and is considered diluted out of the sample with a dilution of 10X or greater. The dilution factor is recorded in Target and used to calculate analyte concentrations and to adjust reporting limits and MDLs. Multiple dilutions are required for some projects. Report multiple dilutions as required. If historical values indicate that the sample needs to be performed at multiple dilutions and all analytes must be reported at the lowest reporting limit possible, the sample extract may be prepared and performed at multiple dilutions without screening.
- G. Manual integrations shall be performed as appropriate. Manual integrations undergo several layers of review and the raw data is flagged. For additional information see SOP QA-010.
- H. Monitor drift by using the internal standard report in target to check the % Difference of internal area response and retention time. Internal area response must be between 50% and 200% of the internal standard response in the continuing calibration verification. Retention time must be within  $\pm 0.5$  minutes. If the criteria are not met reanalyze at a dilution for the affected analytes or report with a narrative.

## QUALITY CONTROL    METHOD BLANK

1. The method blank (extraction blank) is analyzed with each preparatory batch to demonstrate the extraction procedure did not introduce contamination. Run batch QC in the same analytical batch whenever possible. The batch QC must be analyzed with at least one client sample from the prep batch. This blank also establishes the instrument to be free of contamination.
2. No target analytes should be detected in the method blank above one half the reporting limit. If any target analytes are detected, data shall not be reported and samples must be re-extracted and re-analyzed unless the following apply.
  - A. If a target analyte is detected above the reporting limit, data may be reported if the concentration is not greater than 5% of the measured concentration in associated samples. Include a narrative with the data.
  - B. If a target analyte is detected in the method blank but there are no hits in the samples, the data may be reported with a narrative.

## SURROGATES

1. The surrogates are used to verify that each sample was properly extracted and is not adversely affected by the sample matrix. A surrogate is a non-target compound that is chemically similar to the analytes. The surrogates used are listed in the table below. Surrogates are spiked in each sample at the levels listed in Table 1. Some samples use the same extract for scan and SIM analysis. In these cases the surrogates will be spiked at the scan level.
2. Control limits are evaluated after sample analysis. Determine for each sample the project-required limits. If no specific limits are needed, evaluate the recoveries using step 3. If specific limits are needed evaluate using step 4. Evaluate limits as soon as possible to allow necessary re-preps to be performed within holding time.
3. Laboratory derived control limits are listed in the LIMS. One surrogate in each fraction (base-neutral or acid) may have a recovery outside the control limits. When this occurs, report the data with a narrative. If more than one surrogate recovery for each fraction is below the lower control limit, re-extract and re-analyze the sample. If the recoveries are outside control limits in the re-extract sample, then indicate in the case narrative and state that the recovery was outside the control limits due to sample matrix. If re-extraction cannot be performed due to insufficient sample, report the data with a narrative. If the surrogate recovery is above the control limits and the sample results are less than the reporting limit, the data may be reported with a narrative.

4. Project specific recovery limits may apply that must be achieved for the associated samples. See SOP GEN-019. Some projects require reprep and/or reanalysis if any surrogate fails recovery criteria either high or low. Check project criteria before proceeding.

**Table 1**  
**Surrogates**

Surrogates	Scan Conc. mg/L (On column)	SIM Conc mg/L (On column)
<b>Acid Surrogates:</b>		
2-Fluorophenol	100	2
Phenol-d5	100	2
2,4,6-Tribromophenol	100	2
<b>Base - Neutral Surrogates:</b>		
Nitrobenzene-d5	50	1
2-Fluorobiphenyl	50	1
Terphenyl-d14	50	1

#### LABORATORY CONTROL STANDARD (LCS/LCSD)

1. A LCS and LCSD are included in each preparatory batch to demonstrate the system is in control. Routinely the LCS/LCSD will be spiked with the full list of analytes. The control limits for waters and solids are available in the LIMS. Control limits may be project specific and must be achieved for the associated samples. Evaluate the data and perform corrective action based on the project limits. Run batch QC in the same analytical batch whenever possible. The batch QC must be analyzed with at least one client sample from the prep batch. For South Carolina projects, the LCS must be recovered between 70-130%.
2. If a recovery is above the upper control limit and the sample results are below the reporting limit, the data may be reported with a narrative. Check project criteria. Required corrective action may differ in different projects and must be followed for associated samples.
3. If a recovery is below the lower control limit or precision fails, the entire batch must be re-extracted and re-analyzed. If a re-extraction is not possible due to insufficient sample volume, report the data with a narrative.

#### MATRIX SPIKES (MS/MSD)

1. The purpose of the MS/MSD is to assess the performance of the method for a particular sample matrix. The recovery criteria for waters and solids are available in the LIMS and are



typically the same as the LCS recovery criteria. Project specific recovery and RPD limits may apply, and the data evaluated and corrective action performed based on project requirements.

2. The MS/MSD spike includes the full list of analytes.
3. Whenever the MS and/or MSD recoveries are outside the control limits, check that the LCS and LCSD passed criteria to verify the system was in control. If the LCS and LCSD recoveries and precision are acceptable then continue processing the data. If the LCS and/or LCSD failed then follow the procedure in the LCS section.
4. Review data to verify that a lab error has not occurred (wrong spike amount, not spiked) before automatically identifying a failure as matrix interference.
5. If recoveries for both the MS/MSD are outside the control limits and the recoveries are similar, the data is reportable with a narrative stating the LCS recoveries were acceptable. The failure is attributed to sample matrix.
6. Precision is calculated as %RPD. Acceptance criteria are listed in the LIMS. Failures are noted in the narrative.
7. Native sample concentrations may be high in comparison to the spiking concentration and therefore an accurate recovery cannot be calculated. Document this in the case narrative.
8. Spikes may be diluted out in the analysis process. Document this in the case narrative. The spike is diluted out if the dilution is a 10X or greater. MS/MSD are analyzed at the same dilution as the parent sample.

#### SAMPLE

##### RE-EXTRACTION

Samples are to be re-extracted due to failed QC or due to the sample results. When a MB, LCS, or LCSD fails to meet criteria, the entire batch is sent to extractions for re-extraction. If the MS, MSD, or surrogates fail criteria, only the affected samples are sent to extractions. Particular samples may be re-extracted if the sample results do not match historical values, if a sample and a duplicate do not match, or if physical differences are noted in samples. If samples are re-extracted outside of method specified holding times, both analyses are reported. To request sample/batch re-extraction, do the following:

1. If the extract has been analyzed, process the file and load to LIMS.
2. Enter the code "RP" into the analysis code; this will schedule new sample prep.
3. Complete the Re-extraction Request and Tracking Form (attached) and submit a copy to extraction supervisor.

4. When new extracts are brought to the lab, complete the original Re-extraction Request and Tracking Form and report the data appropriately.
5. If additional sample is not available to re-prepare, check with project manager to determine appropriate action to report the available data.
6. If two sets of data will be reported, see login to obtain a re-extracted sample number.

## CALCULATIONS

$$1. \text{ MS \% REC} = \frac{\text{MS Concentration} - \text{Sample Concentration}}{\text{spike added}} \times 100$$

$$2. \text{ MSD \% REC} = \frac{\text{MSD Concentration} - \text{Sample Concentration}}{\text{spike added}} \times 100$$

$$3. \% \text{ RPD} = \frac{\text{MS} - \text{MSD}}{(\text{MS} + \text{MSD}) / 2} \times 100$$

LCS/LCSD results are substituted to calculate %RPD between LCS and LCSD. Results are calculated using the concentration (not percent recovery).

$$4. \text{ Response Factor} = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

$A_s$  = Peak Area of analyte or surrogate

$A_{is}$  = Peak Area of Internal standard

$C_s$  = Concentration of the analyte or surrogate

$C_{is}$  = Concentration of Internal standard

$$5. \text{ Surrogate/LCS Recovery} = \frac{\text{Concentration Found}}{\text{Concentration Added}} \times 100$$

6. Concentration using RF:

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_s) (C_{is}) (D) (V_t)}{(A_{is}) (RF) (V_s)}$$

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_s) (C_{is}) (D) (V_t)}{(A_{is}) (RF) (W_s)}$$

$A_s$  = Area of peak for the analyte in sample

$D$  = Dilution factor

$RF$  = Mean Response factor from initial calibration

$A_{is}$  = Area of internal standard in sample

$C_{is}$  = Concentration of internal standard in  $\mu\text{g/mL}$

$W_s$  = Weight of sample extracted in g

$V_t$  = Total volume of concentrated extract in  $\mu\text{L}$

$V_s$  = Volume of aqueous sample extracted in mL

## 7. Concentration using linear curve fit:

$$\text{Concentration } (\mu\text{g/L}) = [m(A_s/A_{is}) + b]C_{is}D$$

$$\text{Concentration (mg/kg)} = [m(A_s/A_{is}) + b]C_{is}D(5/W_s)$$

m = Inverse of slope

A<sub>s</sub> = Area of peak for the analyte in sample

A<sub>is</sub> = Area of internal standard in sample

b = Intercept of the y-axis

C<sub>is</sub> = Concentration of internal standard in  $\mu\text{g/mL}$

D = Dilution factor

W<sub>s</sub> = Weight of sample extracted

## 8. Concentration of TICS:

$$\text{Concentration (mg/kg)} = \frac{(A_s)(C_{is})(D)}{(A_{is})(1000)}$$

A<sub>s</sub> = Area of peak for the analyte in sample

D = Dilution factor

A<sub>is</sub> = Area of internal standard in sample

C<sub>is</sub> = Concentration of internal standard

W<sub>s</sub> = Weight of sample (if aqueous sample W<sub>s</sub> = 1 and report in  $\mu\text{g/L}$ )

9. % Difference =  $[(RF_I - RF_C) / RF_I] \times 100$  where:

RF<sub>I</sub> = Average response factor from initial calibration

RF<sub>C</sub> = Response factor from current verification check standard

$$10. \% \text{ Drift} = \frac{(\text{Calculated Conc.} - \text{Theoretical Conc.})}{\text{Theoretical Conc.}} \times 100$$

## 11. % RSD = (SD / X) X 100 Where:

RSD = Relative Standard Deviation

X = mean of 5 initial RF's for a compound

SD = Standard Deviation of average RF's for a compound

## TENTATIVELY IDENTIFIED COMPOUNDS

## SEARCH (TIC's)

A TIC search, or library search, may be performed when analyzing samples or sample extracts by GC/MS. A client may request a TIC search for specific compounds that are not calibrated for, or to determine other compounds present in a sample. Generally the client will request a reporting of the 10 or 20 highest concentration "unknowns" in a sample. This is performed by performing a spectral match between an unknown peak and a spectral library. Unknowns are identified based on the spectral data only. In general the retention time is not considered when identifying unknowns. In addition the concentration is an estimate based on the assumption that the response factor of the unknown is 1.0. This may lead to a considerable positive or negative bias in the reported concentration.

The Target method is set-up using the following parameters:

Primary Library - NBS75K.1  
Match Quality Threshold - 85  
Remove Duplicate Matches  
Background Subtraction Method - Average of Front and Rear  
Minimum percent for ID - 10% of nearest Internal Standard Area  
Quantitation by - Area  
RF Mode - Nearest ISTD

The library search does not use a normalization routine. These parameters are set in the Method and are not altered unless approved by a Supervisor or Manager. Note that the selection of peaks and the calculated amount uses the peak area of the nearest internal standard, not the response of quant ion. The area is found in the area.rp report in Target. The amount of an unknown is estimated using an RF factor of 1. To check the calculations performed by Target, use Equation 1.

When performing a library search select Quant Unknown Compounds in Edit Quantitation Parameters and process the data file. The software will identify unknown peaks meeting the set criteria. If the software misses a peak that in the analyst opinion appears to meet the area requirements, the analyst may add an unknown. Select Spectra - Search, then Edit - Edit Unknown. Select the TIC with the best fit (highest quality match). If no match is higher than 50% select the result as "UNKNOWN". If in the analyst's opinion the Target selected best match is incorrect, the match is changed to reflect the best match or is reported as an unknown.

Review the assigned match using the following guidelines:

- 1) Major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.
- 2) The relative intensities of the major ions should agree with the reference spectrum within  $\pm 30\%$ . Major ions that show increased intensity may be due to co-eluting compounds, and ions showing decreased intensity may be due to the background subtraction. When this is encountered examine the spectrum across the peak to determine if co-elution is an issue. If necessary, alter the background subtraction program to best handle the sample matrix.
- 3) Molecular ions present in reference spectrum should be present in sample spectrum (this is usually the heaviest ion).

Save changes and upload results to the LIMS. Only use the Quant Unknown Compound option when a library search is requested. If a library search is added after the file is processed, copy the file using the same file number and append a "T" behind the file number. Process the file and perform the TIC search. Do not reprocess a data file that has already been uploaded to the LIMS.

If a specific analyte is requested to report as a library search, perform the MS library search as described. If the analyte is identified, post the result. If the analyte is not identified, post the TIC file and enter the CAS number, target analyte, and ND in the CAS/Name column of the sample results. Leave the amount column empty.

STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF SEMI-VOLATILE MASS SPEC SAMPLES FOR 8270D	REVISION NO. 7
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#### MANUAL

INTEGRATIONS See GCAL SOP QA-010 for manual integration policy.

#### REPORTING LIMITS

Quantitation limits (PQLs) are defined by the low-level calibration standard. Default reporting limits are employed for reporting and cannot be lower than the PQL. The PQL cannot be lower than the estimated MDL and project specific requirements may define the relationship between the PQL and the MDL that must be achieved for the associated samples. See SOP GEN-019.

#### DATA REVIEW

For data review procedures and checklist, please see SOP QA-002.

#### SOFTWARE

##### TROUBLESHOOTING

The Agilent GC/MS's utilizes Agilent software MSD Chemstation Version D.02.00.275 to control the instrument and transfer data acquisition to Target Data Integration software. Target software is utilized to calculate all data. In the event of software malfunction for the MSD Chemstation, please contact Agilent technical support for troubleshooting help. In the event of software malfunction when using Target, please contact GCAL's IT Director for troubleshooting help.

#### METHOD PERFORMANCE

Method 8250 (the packed column version of Method 8270) was tested by 15 laboratories using organic-free reagent water, drinking water, surface water, and industrial wastewater spiked at six concentrations ranging from 5 to 1,300 ug/L. Single operator accuracy and precision, and method accuracy were found to be directly related to the concentration of the analyte and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 7 of Method 8270C. These values are presented as guidance only and are not intended as absolute acceptance criteria.

Internal control limits are generated for laboratory QC samples including LCS/LCSD, MS/MSD, and surrogates.

#### POLLUTION PREVENTION

See QAPP Section 10.2

#### WASTE MANAGEMENT

See SOP GEN-009

#### IDOC

See SOP QA-014

#### MDL

See SOP QA-009

TABLE 1

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS IN APPROXIMATE  
RETENTION TIME ORDER <sup>a</sup>

Compound	Primary Ion	Secondary Ion(s)
2-Picoline	93	66,92
Aniline	93	66,65
Phenol	94	65,66
Bis(2-chloroethyl) ether	93	63,95
2-Chlorophenol	128	64,130
1,3-Dichlorobenzene	146	148,111
1,4-Dichlorobenzene-d <sub>4</sub> (IS)	152	150,115
1,4-Dichlorobenzene	146	148,111
Benzyl alcohol	108	79,77
1,2-Dichlorobenzene	146	148,111
N-Nitrosomethylethylamine	88	42,43,56
Bis(2-chloroisopropyl) ether	45	77,121
Ethyl carbamate	62	44,45,74
Thiophenol (Benzenethiol)	110	66,109,84
Methyl methanesulfonate	80	79,65,95
N-Nitrosodi-n-propylamine	70	42,101,130
Hexachloroethane	117	201,199
Maleic anhydride	54	98,53,44
Nitrobenzene	77	123,65
Isophorone	82	95,138
N-Nitrosodiethylamine	102	42,57,44,56
2-Nitrophenol	139	109,65
2,4-Dimethylphenol	122	107,121
p-Benzoquinone	108	54,82,80
Bis(2-chloroethoxy)methane	93	95,123
Benzoic acid	122	105,77
2,4-Dichlorophenol	162	164,98
Trimethyl phosphate	110	79,95,109,140
Ethyl methanesulfonate	79	109,97,45,65
1,2,4-Trichlorobenzene	180	182,145
Naphthalene-d <sub>8</sub> (IS)	136	68
Naphthalene	128	129,127
Hexachlorobutadiene	225	223,227
Tetraethyl pyrophosphate	99	155,127,81,109
Diethyl sulfate	139	45,59,99,111,125
4-Chloro-3-methylphenol	107	144,142
2-Methylnaphthalene	142	141
2-Methylphenol	107	108,77,79,90
Hexachloropropene	213	211,215,117,106,141
Hexachlorocyclopentadiene	237	235,272
N-Nitrosopyrrolidine	100	41,42,68,69
Acetophenone	105	71,51,120
3/4-Methylphenol <sup>b</sup>	107	108,77,79,90

TABLE 1  
(continued)

Compound	Primary Ion	Secondary Ion(s)
2,4,6-Trichlorophenol	196	198,200
o-Toluidine	106	107,77,51,79
2-Chloronaphthalene	162	127,164
N-Nitrosopiperidine	114	42,55,56,41
1,4-Phenylenediamine	108	80,53,54,52
1-Chloronaphthalene	162	127,164
2-Nitroaniline	65	92,138
5-Chloro-2-methylaniline	106	141,140,77,89
Dimethyl phthalate	163	194,164
Acenaphthylene	152	151,153
2,6-Dinitrotoluene	165	63,89
Phthalic anhydride	104	76,50,148
o-Anisidine	108	80,123,52
3-Nitroaniline	138	108,92
Acenaphthene-d <sub>10</sub> (IS)	164	162,160
Acenaphthene	154	153,152
2,4-Dinitrophenol	184	63,154
2,6-Dinitrophenol	162	164,126,98,63
4-Chloroaniline	127	129,65,92
Isosafrole	162	131,104,77,51
Dibenzofuran	168	139
2,4-Diaminotoluene	121	122,94,77,104
2,4-Dinitrotoluene	165	63,89
4-Nitrophenol	139	109,65
2-Naphthylamine	143	115,116
1,4-Naphthoquinone	158	104,102,76,50,130
p-Cresidine	122	94,137,77,93
Dichlorovos	109	185,79,145
Diethyl phthalate	149	177,150
Fluorene	166	165,167
2,4,5-Trimethylaniline	120	135,134,91,77
N-Nitrosodi-n-butylamine	84	57,41,116,158
4-Chlorophenyl phenyl ether	204	206,141
Hydroquinone	110	81,53,55
4,6-Dinitro-2-methylphenol	198	51,105
Resorcinol	110	81,82,53,69
N-Nitrosodiphenylamine	169	168,167
Safrole	162	104,77,103,135
Hexamethyl phosphoramidate	135	44,179,92,42
3-(Chloromethyl)pyridine hydrochloride	92	127,129,65,39
Diphenylamine	169	168,167
1,2,4,5-Tetrachlorobenzene	216	214,179,108,143,218
1-Naphthylamine	143	115,89,63
1-Acetyl-2-thiourea	118	43,42,76
4-Bromophenyl phenyl ether	248	250,141



TABLE 1  
(continued)

Compound	Primary Ion	Secondary Ion(s)
Toluene diisocyanate	174	145,173,146,132,91
2,4,5-Trichlorophenol	196	198,97,132,99
Hexachlorobenzene	284	142,249
Nicotine	84	133,161,162
Pentachlorophenol	266	264,268
5-Nitro-o-toluidine	152	77,79,106,94
Thionazine	107	96,97,143,79,68
4-Nitroaniline	138	65,108,92,80,39
Phenanthrene-d <sub>10</sub> (IS)	188	94,80
Phenanthrene	178	179,176
Anthracene	178	176,179
1,4-Dinitrobenzene	168	75,50,76,92,122
Mevinphos	127	192,109,67,164
Naled	109	145,147,301,79,189
1,3-Dinitrobenzene	168	76,50,75,92,122
Diallate (cis or trans)	86	234,43,70
1,2-Dinitrobenzene	168	50,63,74
Diallate (trans or cis)	86	234,43,70
Pentachlorobenzene	250	252,108,248,215,254
5-Nitro-o-anisidine	168	79,52,138,153,77
Pentachloronitrobenzene	237	142,214,249,295,265
4-Nitroquinoline-1-oxide	174	101,128,75,116
Di-n-butyl phthalate	149	150,104
2,3,4,6-Tetrachlorophenol	232	131,230,166,234,168
Dihydrosaffrole	135	64,77
Demeton-O	88	89,60,61,115,171
Fluoranthene	202	101,203
1,3,5-Trinitrobenzene	75	74,213,120,91,63
Dicrotophos	127	67,72,109,193,237
Benzidine	184	92,185
Trifluralin	306	43,264,41,290
Bromoxynil	277	279,88,275,168
Pyrene	202	200,203
Monocrotophos	127	192,67,97,109
Phorate	75	121,97,93,260
Sulfallate	188	88,72,60,44
Demeton-S	88	60,81,89,114,115
Phenacetin	108	180,179,109,137,80
Dimethoate	87	93,125,143,229
Phenobarbital	204	117,232,146,161
Carbofuran	164	149,131,122
Octamethyl pyrophosphoramide	135	44,199,286,153,243
4-Aminobiphenyl	169	168,170,115
Dioxathion	97	125,270,153
Terbufos	231	57,97,153,103

TABLE 1  
(continued)

Compound	Primary Ion	Secondary Ion(s)
$\alpha,\alpha$ -Dimethylphenylamine	58	91,65,134,42
Pronamide	173	175,145,109,147
Aminoazobenzene	197	92,120,65,77
Dichlone	191	163,226,228,135,193
Dinoseb	211	163,147,117,240
Disulfoton	88	97,89,142,186
Fluchloralin	306	63,326,328,264,65
Mexacarbate	165	150,134,164,222
4,4'-Oxydianiline	200	108,171,80,65
Butyl benzyl phthalate	149	91,206
4-Nitrobiphenyl	199	152,141,169,151
Phosphamidon	127	264,72,109,138
2-Cyclohexyl-4,6-Dinitrophenol	231	185,41,193,266
Methyl parathion	109	125,263,79,93
Carbaryl	144	115,116,201
Dimethylaminoazobenzene	225	120,77,105,148,42
Propylthiouracil	170	142,114,83
Benz(a)anthracene	228	229,226
Chrysene-d <sub>12</sub> (IS)	240	120,236
3,3'-Dichlorobenzidine	252	254,126
Chrysene	228	226,229
Malathion	173	125,127,93,158
Kepone	272	274,237,178,143,270
Fenthion	278	125,109,169,153
Parathion	109	97,291,139,155
Anilazine	239	241,143,178,89
Bis(2-ethylhexyl) phthalate	149	167,279
3,3'-Dimethylbenzidine	212	106,196,180
Carbophenothion	157	97,121,342,159,199
5-Nitroacenaphthene	199	152,169,141,115
Methapyrilene	97	50,191,71
Isodrin	193	66,195,263,265,147
Captan	79	149,77,119,117
Chlorfenvinphos	267	269,323,325,295
Crotoxyphos	127	105,193,166
Phosmet	160	77,93,317,76
EPN	157	169,185,141,323
Tetrachlorvinphos	329	109,331,79,333
Di-n-octyl phthalate	149	167,43
2-Aminoanthraquinone	223	167,195
Barban	222	51,87,224,257,153
Aramite	185	191,319,334,197,321
Benzo(b)fluoranthene	252	253,125
Nitrofen	283	285,202,139,253
Benzo(k)fluoranthene	252	253,125

TABLE 1  
(continued)

Compound	Primary Ion	Secondary Ion(s)
Chlorobenzilate	251	139,253,111,141
Fensulfothion	293	97,308,125,292
Ethion	231	97,153,125,121
Diethylstilbestrol	268	145,107,239,121,159
Famphur	218	125,93,109,217
Tri-p-tolyl phosphate <sup>c</sup>	368	367,107,165,198
Benzo(a)pyrene	252	253,125
Perylene-d <sub>12</sub> (IS)	264	260,265
7,12-Dimethylbenz(a)anthracene	256	241,239,120
5,5-Diphenylhydantoin	180	104,252,223,209
Captafol	79	77,80,107
Dinocap	69	41,39
Methoxychlor	227	228,152,114,274,212
2-Acetylaminofluorene	181	180,223,152
4,4'-Methylenebis(2-chloroaniline)	231	266,268,140,195
3,3'-Dimethoxybenzidine	244	201,229
3-Methylcholanthrene	268	252,253,126,134,113
Phosalone	182	184,367,121,379
Azinphos-methyl	160	132,93,104,105
Leptophos	171	377,375,77,155,379
Mirex	272	237,274,270,239,235
Tris(2,3-dibromopropyl) phosphate	201	137,119,217,219,199
Dibenz(a,j)acridine	279	280,277,250
Mestranol	277	310,174,147,242
Coumaphos	362	226,210,364,97,109
Indeno(1,2,3-cd)pyrene	276	138,277
Dibenz(a,h)anthracene	278	139,279
Benzo(g,h,i)perylene	276	138,277
1,2:4,5-Dibenzopyrene	302	151,150,300
Strychnine	334	334,335,333
Piperonyl sulfoxide	162	135,105,77
Hexachlorophene	196	198,209,211,406,408
Aldrin	66	263,220
Aroclor 1016	222	260,292
Aroclor 1221	190	224,260
Aroclor 1232	190	224,260
Aroclor 1242	222	256,292
Aroclor 1248	292	362,326
Aroclor 1254	292	362,326
Aroclor 1260	360	362,394
α-BHC	183	181,109
β-BHC	181	183,109
δ-BHC	183	181,109
γ-BHC (Lindane)	183	181,109
4,4'-DDD	235	237,165

TABLE 1  
(continued)

Compound	Primary Ion	Secondary Ion(s)
4,4'-DDE	246	248,176
4,4'-DDT	235	237,165
Dieldrin	79	263,279
1,2-Diphenylhydrazine	77	105,182
Endosulfan I	195	339,341
Endosulfan II	337	339,341
Endosulfan sulfate	272	387,422
Endrin	263	82,81
Endrin aldehyde	67	345,250
Endrin ketone	317	67,319
2-Fluorobiphenyl (surr)	172	171
2-Fluorophenol (surr)	112	64
Heptachlor	100	272,274
Heptachlor epoxide	353	355,351
Nitrobenzene-d <sub>5</sub> (surr)	82	128,54
N-Nitrosodimethylamine	42	74,44
Phenol-d <sub>6</sub> (surr)	99	42,71
Terphenyl-d <sub>14</sub> (surr)	244	122,212
2,4,6-Tribromophenol (surr)	330	332,141
Toxaphene	159	231,233

IS = internal standard

surr = surrogate

<sup>a</sup> The data presented are representative of DB-5 type analytical columns

<sup>b</sup> Compounds cannot be separated for quantitation

<sup>c</sup> Substitute for the non-specific mixture, tricresyl phosphate

### Appendix III

#### Control Limits

#### Gulf Coast Analytical Laboratories

Queue: MSSV

Method: SW-846 8270D

LCS			
Analyte	Name	LCL-UCL	Matrix
126-68-1	0,0,0-Triethylphosphorothioate	60-120	W
95-94-3	1,2,4,5-Tetrachlorobenzene	60-120	W
120-82-1	1,2,4-Trichlorobenzene	30-120	W
95-50-1	1,2-Dichlorobenzene	27-120	W
122-66-7	1,2Diphenylhydrazine/Azobenzen	60-120	W
99-35-4	1,3,5-Trinitrobenzene	60-120	W
541-73-1	1,3-Dichlorobenzene	25-120	W
99-65-0	1,3-Dinitrobenzene	60-120	W
106-46-7	1,4-Dichlorobenzene	22-120	W
100-25-4	1,4-Dinitrobenzene	60-120	W
123-91-1	1,4-Dioxane	1-120	W
130-15-4	1,4-Naphthoquinone	60-120	W
106-50-3	1,4-Phenylenediamine	60-120	W
111-29-5	1,5-Pentanediol	60-120	W
629-11-8	1,6-Hexanediol	60-120	W
90-12-0	1-Methylnaphthalene	38-120	W
134-32-7	1-Naphthylamine	60-120	W
2687-25-4	2,3 and/or 3,4-Diaminotoluene	4-130	W
58-90-2	2,3,4,6-Tetrachlorophenol	60-120	W
15950-66-0	2,3,4-Trichlorophenol	40-160	W
935-95-5	2,3,5,6-Tetrachlorophenol	40-160	W
MSSV35	2,4 and/or 2,5-Dichlorophenol	55-120	W
95-80-7M	2,4 and/or 2,6-Diaminotoluene	4-130	W
95-95-4	2,4,5-Trichlorophenol	60-120	W
88-06-2	2,4,6-Trichlorophenol	59-120	W
120-83-2	2,4-Dichlorophenol	55-120	W
105-67-9	2,4-Dimethylphenol	14-122	W
51-28-5	2,4-Dinitrophenol	18-137	W
121-14-2	2,4-Dinitrotoluene	37-138	W
87-65-0	2,6-Dichlorophenol	48-120	W
606-20-2	2,6-Dinitrotoluene	56-128	W
53-96-3	2-Acetylaminofluorene	60-120	W
91-58-7	2-Chloronaphthalene	48-120	W
95-57-8	2-Chlorophenol	42-120	W
91-57-6	2-Methylnaphthalene	40-120	W
91-59-8	2-Naphthylamine	60-120	W
88-74-4	2-Nitroaniline	48-120	W
88-75-5	2-Nitrophenol	59-120	W
109-06-8	2-Picoline	60-120	W

MSSV34	3 and/or 4-Chlorophenol	40-160	W
91-94-1	3,3'-Dichlorobenzidine	51-154	W
119-93-7	3,3'-Dimethylbenzidine	60-120	W
609-19-8	3,4,5-Trichlorophenol	40-160	W
95-77-2	3,4-Dichlorophenol	40-160	W
56-49-5	3-Methylcholanthrene	60-120	W
99-09-2	3-Nitroaniline	34-120	W
101-14-4	4,4'-Methylenebis(2-chloroanili	60-120	W
101-77-9	4,4'-Methylenedianiline (MDA)	20-120	W
534-52-1	4,6-Dinitro-2-methylphenol	49-120	W
92-67-1	4-Aminobiphenyl	60-120	W
101-55-3	4-Bromophenyl phenyl ether	61-120	W
59-50-7	4-Chloro-3-methylphenol	44-120	W
106-47-8	4-Chloroaniline	30-120	W
7005-72-3	4-Chlorophenyl phenyl ether	52-120	W
100-01-6	4-Nitroaniline	38-120	W
100-02-7	4-Nitrophenol	3-120	W
56-57-5	4-Nitroquinoline-1-oxide	60-120	W
99-55-8	5-Nitro-o-toluidine	60-120	W
57-97-6	7,12-Dimethylbenz(a)anthracene	60-120	W
83-32-9	Acenaphthene	52-120	W
208-96-8	Acenaphthylene	55-120	W
98-86-2	Acetophenone	60-124	W
79-06-1	Acrylamide	60-120	W
62-53-3	Aniline	19-124	W
120-12-7	Anthracene	58-120	W
140-57-8	Aramite	60-120	W
1912-24-9	Atrazine (Aatrex)	39-148	W
100-52-7	Benzaldehyde	40-118	W
92-87-5	Benzidine	10-120	W
56-55-3	Benzo(a)anthracene	56-120	W
50-32-8	Benzo(a)pyrene	56-120	W
205-99-2	Benzo(b)fluoranthene	55-120	W
191-24-2	Benzo(g,h,i)perylene	44-132	W
207-08-9	Benzo(k)fluoranthene	49-121	W
65-85-0	Benzoic acid	5-120	W
100-51-6	Benzyl alcohol	32-120	W
92-52-4	Biphenyl	39-122	W
111-91-1	Bis(2-Chloroethoxy)methane	56-120	W
111-44-4	Bis(2-Chloroethyl)ether	37-120	W
108-60-1	Bis(2-Chloroisopropyl)ether	47-120	W
117-81-7	Bis(2-Ethylhexyl)phthalate	56-132	W
85-68-7	Butyl benzyl phthalate	62-122	W
105-60-2	Caprolactam	10-120	W
86-74-8	Carbazole	47-120	W
510-15-6	Chlorobenzilate	60-120	W
218-01-9	Chrysene	58-120	W

1319-77-3	Cresols	60-120	W
108-93-0	Cyclohexanol	60-120	W
84-74-2	Di-n-butyl phthalate	62-122	W
117-84-0	Di-n-octyl phthalate	56-133	W
2303-16-4	Diallate (Avadex)	60-120	W
53-70-3	Dibenz(a,h)anthracene	50-138	W
132-64-9	Dibenzofuran	54-120	W
77-73-6	Dicyclopentadiene	10-120	W
GCSV-00-4	Diesel Range Organics	47-120	W
84-66-2	Diethyl phthalate	56-120	W
60-51-5	Dimethoate	60-120	W
131-11-3	Dimethyl phthalate	59-120	W
88-85-7	Dinoseb	60-120	W
122-39-4	Diphenylamine	60-120	W
928-72-3	Disodium Iminoacetate	10-120	W
298-04-4	Disulfoton	60-120	W
56-38-2	Ethyl Parathion	60-120	W
62-50-0	Ethyl methanesulfonate	60-120	W
52-85-7	Famphur	60-120	W
206-44-0	Fluoranthene	55-120	W
86-73-7	Fluorene	54-120	W
118-74-1	Hexachlorobenzene	61-120	W
87-68-3	Hexachlorobutadiene	17-120	W
77-47-4	Hexachlorocyclopentadiene	16-120	W
67-72-1	Hexachloroethane	21-120	W
70-30-4	Hexachlorophene	60-120	W
1888-71-7	Hexachloropropene	60-120	W
142-62-1	Hexanoic Acid	60-120	W
193-39-5	Indeno(1,2,3-cd)pyrene	43-133	W
465-73-6	Isodrin	60-120	W
78-59-1	Isophorone	53-120	W
120-58-1	Isosafrole	60-120	W
143-50-0	Kepone	60-120	W
108-31-6	Maleic anhydride	60-120	W
91-80-5	Methapyriline	60-120	W
66-27-3	Methyl methanesulfonate	60-120	W
298-00-0	Methyl parathion	60-120	W
91-20-3	Naphthalene	44-120	W
98-95-3	Nitrobenzene	53-120	W
GCSV-00-44	Oil Range Organics	47-120	W
608-93-5	Pentachlorobenzene	60-120	W
76-01-7	Pentachloroethane	60-120	W
82-68-8	Pentachloronitrobenzene	60-120	W
87-86-5	Pentachlorophenol	25-158	W
109-52-4	Pentanoic Acid	60-120	W
62-44-2	Phenacetin	60-120	W
85-01-8	Phenanthrene	58-120	W



108-95-2	Phenol	16-120	W
298-02-2	Phorate	60-120	W
88-99-3	Phthalic acid/anhydride	60-120	W
23950-58-5	Pronamide	60-120	W
129-00-0	Pyrene	54-120	W
110-86-1	Pyridine	2-120	W
91-22-5	Quinoline	60-120	W
94-59-7	Safrole	60-120	W
126-33-0	Sulfolane	60-120	W
3689-24-5	Sulfotep	60-120	W
297-97-2	Thionazine	60-120	W
95-80-7	Toluene diamine	4-120	W
I-317	Toluene diisocyanate	4-120	W
SVOA-001	Total Methyl-naphthalene	60-120	W
126-73-8	Tributyl Phosphate	60-120	W
126-72-7	Tris(2,3-dibromopropyl)phosph	60-120	W
122-09-8	a,a-Dimethylphenethylamine	60-120	W
1319-77-3MP	m,p-Cresol	24-120	W
924-16-3	n-Nitrosodi-n-butylamine	60-120	W
621-64-7	n-Nitrosodi-n-propylamine	47-120	W
55-18-5	n-Nitrosodiethylamine	60-120	W
62-75-9	n-Nitrosodimethylamine	12-120	W
86-30-6	n-Nitrosodiphenylamine	58-121	W
10595-95-6	n-Nitrosomethylethylamine	60-120	W
59-89-2	n-Nitrosomorpholine	60-120	W
100-75-4	n-Nitrosopiperidine	60-120	W
930-55-2	n-Nitrosopyrrolidine	60-120	W
95-53-4	o and/or p - Toluidine	60-120	W
95-48-7	o-Cresol	31-120	W
60-11-7	p-(Dimethylamino)azobenzene	60-120	W
106-51-4	p-Benzoquinone	60-120	W

### Control Limits

#### Gulf Coast Analytical Laboratories

Queue: MSSV

Method: SW-846 8270D

LCS			
Analyte	Name	LCL-UCL	Matrix
126-68-1	0,0,0-Triethylphosphorothioate	60-120	S
95-94-3	1,2,4,5-Tetrachlorobenzene	60-120	S
120-82-1	1,2,4-Trichlorobenzene	46-120	S
95-50-1	1,2-Dichlorobenzene	44-120	S
122-66-7	1,2Diphenylhydrazine/Azobenzen	49-120	S
99-35-4	1,3,5-Trinitrobenzene	60-120	S
541-73-1	1,3-Dichlorobenzene	40-120	S
99-65-0	1,3-Dinitrobenzene	60-120	S

106-46-7	1,4-Dichlorobenzene	42-120	S
100-25-4	1,4-Dinitrobenzene	60-120	S
123-91-1	1,4-Dioxane	10-130	S
130-15-4	1,4-Naphthoquinone	60-120	S
106-50-3	1,4-Phenylenediamine	60-120	S
111-29-5	1,5-Pentanediol	60-120	S
629-11-8	1,6-Hexanediol	60-120	S
90-12-0	1-Methylnaphthalene	37-120	S
134-32-7	1-Naphthylamine	60-120	S
2687-25-4	2,3 and/or 3,4-Diaminotoluene	4-130	S
58-90-2	2,3,4,6-Tetrachlorophenol	60-120	S
95-80-7M	2,4 and/or 2,6-Diaminotoluene	10-130	S
95-95-4	2,4,5-Trichlorophenol	47-120	S
88-06-2	2,4,6-Trichlorophenol	46-120	S
120-83-2	2,4-Dichlorophenol	47-120	S
105-67-9	2,4-Dimethylphenol	47-120	S
51-28-5	2,4-Dinitrophenol	14-120	S
121-14-2	2,4-Dinitrotoluene	45-120	S
87-65-0	2,6-Dichlorophenol	60-120	S
606-20-2	2,6-Dinitrotoluene	47-120	S
934-34-9	2-(3H) Benzothiazolone	30-120	S
149-30-4	2-(3H)Benzothiazolethione	30-120	S
53-96-3	2-Acetylaminofluorene	60-120	S
91-58-7	2-Chloronaphthalene	52-120	S
95-57-8	2-Chlorophenol	48-120	S
91-57-6	2-Methylnaphthalene	43-120	S
91-59-8	2-Naphthylamine	60-120	S
88-74-4	2-Nitroaniline	44-120	S
88-75-5	2-Nitrophenol	49-120	S
109-06-8	2-Picoline	60-120	S
91-94-1	3,3'-Dichlorobenzidine	35-120	S
119-93-7	3,3'-Dimethylbenzidine	60-120	S
56-49-5	3-Methylcholanthrene	60-120	S
99-09-2	3-Nitroaniline	29-120	S
101-14-4	4,4'-Methylenebis(2-chloroanili	60-120	S
534-52-1	4,6-Dinitro-2-methylphenol	29-120	S
92-67-1	4-Aminobiphenyl	60-120	S
101-55-3	4-Bromophenyl phenyl ether	51-125	S
59-50-7	4-Chloro-3-methylphenol	46-120	S
106-47-8	4-Chloroaniline	20-120	S
7005-72-3	4-Chlorophenyl phenyl ether	50-120	S
101-77-9	4,4'-Methylenedianiline (MDA)	20-120	S
100-01-6	4-Nitroaniline	32-120	S
100-02-7	4-Nitrophenol	32-120	S
56-57-5	4-Nitroquinoline-1-oxide	60-120	S
99-55-8	5-Nitro-o-toluidine	60-120	S
57-97-6	7,12-Dimethylbenz(a)anthracene	60-120	S

83-32-9	Acenaphthene	50-120	S
208-96-8	Acenaphthylene	53-120	S
98-86-2	Acetophenone	49-120	S
79-06-1	Acrylamide	60-120	S
62-53-3	Aniline	21-131	S
120-12-7	Anthracene	52-120	S
140-57-8	Aramite	60-120	S
1912-24-9	Atrazine (Aatrex)	43-150	S
100-52-7	Benzaldehyde	25-127	S
92-87-5	Benzidine	8-120	S
56-55-3	Benzo(a)anthracene	48-120	S
50-32-8	Benzo(a)pyrene	44-120	S
205-99-2	Benzo(b)fluoranthene	31-130	S
191-24-2	Benzo(g,h,i)perylene	29-134	S
207-08-9	Benzo(k)fluoranthene	36-122	S
65-85-0	Benzoic acid	14-124	S
95-16-9	Benzothiazole	30-120	S
100-51-6	Benzyl alcohol	47-120	S
92-52-4	Biphenyl	46-120	S
111-91-1	Bis(2-Chloroethoxy)methane	51-120	S
111-44-4	Bis(2-Chloroethyl)ether	46-120	S
108-60-1	Bis(2-Chloroisopropyl)ether	46-120	S
117-81-7	Bis(2-Ethylhexyl)phthalate	46-129	S
85-68-7	Butyl benzyl phthalate	46-130	S
105-60-2	Caprolactam	34-120	S
86-74-8	Carbazole	47-120	S
510-15-6	Chlorobenzilate	60-120	S
218-01-9	Chrysene	51-120	S
1319-77-3	Cresols	46-120	S
108-93-0	Cyclohexanol	60-120	S
84-74-2	Di-n-butyl phthalate	50-120	S
117-84-0	Di-n-octyl phthalate	41-122	S
2303-16-4	Diallate (Avadex)	60-120	S
53-70-3	Dibenz(a,h)anthracene	27-129	S
132-64-9	Dibenzofuran	50-120	S
77-73-6	Dicyclopentadiene	60-120	S
GCSV-00-4	Diesel Range Organics	50-124	S
84-66-2	Diethyl phthalate	36-120	S
60-51-5	Dimethoate	60-120	S
617-94-7	Dimethyl benzyl alcohol	60-120	S
131-11-3	Dimethyl phthalate	50-120	S
88-85-7	Dinoseb	60-120	S
101-84-8	Diphenyl Ether	60-120	S
122-39-4	Diphenylamine	60-120	S
928-72-3	Disodium Iminoacetate	40-160	S
298-04-4	Disulfoton	60-120	S
56-38-2	Ethyl Parathion	60-120	S

62-50-0	Ethyl methanesulfonate	60-120	S
52-85-7	Famphur	60-120	S
206-44-0	Fluoranthene	39-120	S
86-73-7	Fluorene	48-120	S
118-74-1	Hexachlorobenzene	48-120	S
87-68-3	Hexachlorobutadiene	46-120	S
77-47-4	Hexachlorocyclopentadiene	23-121	S
67-72-1	Hexachloroethane	40-120	S
70-30-4	Hexachlorophene	60-120	S
1888-71-7	Hexachloropropene	60-120	S
142-62-1	Hexanoic Acid	60-120	S
193-39-5	Indeno(1,2,3-cd)pyrene	43-132	S
465-73-6	Isodrin	60-120	S
78-59-1	Isophorone	49-120	S
120-58-1	Isosafrole	60-120	S
143-50-0	Kepone	60-120	S
108-31-6	Maleic anhydride	60-120	S
91-80-5	Methapyriline	60-120	S
66-27-3	Methyl methanesulfonate	60-120	S
298-00-0	Methyl parathion	60-120	S
91-20-3	Naphthalene	48-120	S
98-95-3	Nitrobenzene	45-120	S
GCSV-00-44	Oil Range Organics	47-120	S
608-93-5	Pentachlorobenzene	60-120	S
76-01-7	Pentachloroethane	60-120	S
82-68-8	Pentachloronitrobenzene	60-120	S
87-86-5	Pentachlorophenol	30-124	S
109-52-4	Pentanoic Acid	60-120	S
62-44-2	Phenacetin	60-120	S
85-01-8	Phenanthrene	53-120	S
108-95-2	Phenol	42-120	S
298-02-2	Phorate	60-120	S
88-99-3	Phthalic acid/anhydride	60-120	S
23950-58-5	Pronamide	60-120	S
129-00-0	Pyrene	38-136	S
110-86-1	Pyridine	11-120	S
91-22-5	Quinoline	60-120	S
94-59-7	Safrole	60-120	S
126-33-0	Sulfolane	60-120	S
3689-24-5	Sulfotep	60-120	S
297-97-2	Thionazine	60-120	S
95-80-7	Toluene diamine	10-130	S
I-317	Toluene diisocyanate	4-120	S
GCSV-05-9	Total Hydrocarbons (JP8)	50-150	S
SVOA-001	Total Methylnaphthalene	37-120	S
126-73-8	Tributyl Phosphate	60-120	S
126-72-7	Tris(2,3-dibromopropyl)phosph	60-120	S

122-09-8	a,a-Dimethylphenethylamine	60-120	S
1319-77-3MP	m,p-Cresol	46-120	S
105-59-9	n-Methyldiethanolamine	60-120	S
924-16-3	n-Nitrosodi-n-butylamine	60-120	S
621-64-7	n-Nitrosodi-n-propylamine	46-120	S
55-18-5	n-Nitrosodiethylamine	60-120	S
62-75-9	n-Nitrosodimethylamine	34-126	S
86-30-6	n-Nitrosodiphenylamine	54-125	S
10595-95-6	n-Nitrosomethylethylamine	60-120	S
59-89-2	n-Nitrosomorpholine	60-120	S
100-75-4	n-Nitrosopiperidine	60-120	S
930-55-2	n-Nitrosopyrrolidine	60-120	S
95-53-4	o and/or p - Toluidine	60-120	S
95-48-7	o-Cresol	46-120	S
60-11-7	p-(Dimethylamino)azobenzene	60-120	S
106-51-4	p-Benzoquinone	60-120	S

## Appendix IV

0,0,0-Triethylphosphorothioate	5-Nitro-o-toluidine	Isodrin
1,2,4,5-Tetrachlorobenzene	7,12-Dimethylbenz(a)anthracene	Isophorone
1,2,4-Trichlorobenzene	Acenaphthene	Isosafrole
1,2-Dichlorobenzene	Acenaphthylene	Kepone
1,2Diphenylhydrazine/Azobenzen	Acetophenone	Maleic anhydride
1,3,5-Trinitrobenzene	Acrylamide	Methapyriline
1,3-Dichlorobenzene	Aniline	Methyl methanesulfonate
1,3-Dinitrobenzene	Anthracene	Methyl parathion
1,4-Dichlorobenzene	Aramite	Naphthalene
1,4-Dinitrobenzene	Atrazine (Aatrex)	Nitrobenzene
1,4-Dioxane	Benzaldehyde	Pentachlorobenzene
1,4-Naphthoquinone	Benzidine	Pentachloroethane
1,4-Phenylenediamine	Benzo(a)anthracene	Pentachloronitrobenzene
1,5-Pentanediol	Benzo(a)pyrene	Pentachlorophenol
1,6-Hexanediol	Benzo(b)fluoranthene	Pentanoic Acid
1-Methylnaphthalene	Benzo(g,h,i)perylene	Phenacetin
1-Naphthylamine	Benzo(k)fluoranthene	Phenanthrene
2,3 and/or 3,4-Diaminotoluene	Benzoic acid	Phenol
2,3,4,6-Tetrachlorophenol	Benzyl alcohol	Phorate
2,3,4-Trichlorophenol	Biphenyl	Phthalic acid/anhydride
2,3,5,6-Tetrachlorophenol	Bis(2-Chloroethoxy)methane	Pronamide
2,4 and/or 2,5-Dichlorophenol	Bis(2-Chloroethyl)ether	Pyrene
2,4 and/or 2,6-Diaminotoluene	Bis(2-Chloroisopropyl)ether	Pyridine
2,4,5-Trichlorophenol	Bis(2-Ethylhexyl)phthalate	Quinoline
2,4,6-Trichlorophenol	Butyl benzyl phthalate	Safrole
2,4-Dichlorophenol	Caprolactam	Sulfolane
2,4-Dimethylphenol	Carbazole	Sulfotep
2,4-Dinitrophenol	Chlorobenzilate	Thionazine
2,4-Dinitrotoluene	Chrysene	Toluene diamine
2,6-Dichlorophenol	Cresols	Toluene diisocyanate
2,6-Dinitrotoluene	Cyclohexanol	Total Methylnaphthalene
2-Acetylaminofluorene	Di-n-butyl phthalate	Tributyl Phosphate
2-Chloronaphthalene	Di-n-octyl phthalate	Tris(2,3-dibromopropyl)phosph
2-Chlorophenol	Diallate (Avadex)	a,a-Dimethylphenethylamine
2-Methylnaphthalene	Dibenz(a,h)anthracene	m,p-Cresol
2-Naphthylamine	Dibenzofuran	n-Nitrosodi-n-butylamine
2-Nitroaniline	Dicyclopentadiene	n-Nitrosodi-n-propylamine
2-Nitrophenol	Diethyl phthalate	n-Nitrosodiethylamine
2-Picoline	Dimethoate	n-Nitrosodimethylamine
3 and/or 4-Chlorophenol	Dimethyl phthalate	n-Nitrosodiphenylamine
3,3'-Dichlorobenzidine	Dinoseb	n-Nitrosomethylethylamine
3,3'-Dimethylbenzidine	Diphenylamine	n-Nitrosomorpholine
3,4,5-Trichlorophenol	Disodium Iminoacetate	n-Nitrosopiperidine
3,4-Dichlorophenol	Disulfoton	n-Nitrosopyrrolidine
3-Methylcholanthrene	Ethyl Parathion	o and/or p - Toluidine
3-Nitroaniline	Ethyl methanesulfonate	o-Cresol
4,4'-Methylenebis(2-chloroanili	Famphur	p-(Dimethylamino)azobenzene
4,6-Dinitro-2-methylphenol	Fluoranthene	p-Benzoquinone
4-Aminobiphenyl	Fluorene	
4-Bromophenyl phenyl ether	Hexachlorobenzene	
4-Chloro-3-methylphenol	Hexachlorobutadiene	
4-Chloroaniline	Hexachlorocyclopentadiene	
4-Chlorophenyl phenyl ether	Hexachloroethane	
4,4'-Methylenedianiline (MDA)	Hexachlorophene	
4-Nitroaniline	Hexachloropropene	
4-Nitrophenol	Hexanoic Acid	
4-Nitroquinoline-1-oxide	Indeno(1,2,3-cd)pyrene	

## APPENDIX A

### GLOSSARY

ACCEPTANCE LIMITS - data quality limits specified by the test method or generated by the laboratory. Specified limits placed on an item, process, or service defined in the requirement document.

ACCURACY - a measure of how close a measured value is to a known true value. Accuracy is assessed by means of reference samples and percent recoveries of spiked samples. The degree of agreement between an observed value and an accepted reference value. Accuracy includes combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

ALIQOT - a discrete, measure, or representative portion of a sample taken for analysis.

ANALYTE - the chemical element or compound an analyst seeks to determine; the chemical element of interest.

ANALYTICAL BATCH - the basic unit for analytical quality control, defined as samples that are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. Samples in each batch should be of similar composition (e.g., groundwater, sludge, ash).

ANALYTICAL SAMPLE - any solution or media introduced into an instrument on which an analysis is performed, excluding instrument calibration, initial calibration verification, initial calibration blank, continuing calibration verification, and continuing calibration blank. The following are all analytical samples: undiluted and diluted samples (EPA and non-EPA), predigestion spike samples, duplicate samples, serial dilution samples, analytical spike samples, post digestion spike samples, interference check samples (ICS), CRDL standard for AA(CRA), CRDL standard for ICP(CRI), laboratory control sample(LCS), preparation blank(PB), and linear range analysis sample(LRS).

ANALYST - the designated individual who performs the "hands on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

AREA UNITS - a term used in gas chromatography that indicates the peak area of a compound exiting a chromatographic column. The size or area of the peak is proportional to the amount of analyte in the sample.

ATOMIC ABSORPTION (AA) - a technique for analyzing metal using an element-specific lamp that emits a characteristic light spectrum. A sample is heated in a flame or graphite furnace and the light beam is passed through it. When the sample absorbs light, an energy loss is detected and is translated into a concentration of metal in the sample. This technique detects one metal at a time.

AUDIT - a systematic check to determine the quality of some function or activity. Two basic types are performance audits and system audits. Perform audits involve a quantitative comparison of the laboratory's results to those of a proficiency sample containing known concentration of analytes. A system audit is a qualitative evaluation that normally consists of an onsite review of a lab's quality assurance system and physical facilities.



BACKGROUND CORRECTION - a technique usually employed relative to metals analysis, which compensates for variable background contribution to the instrument signal in the determination of trace elements.

BATCH - environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A batch is composed of a maximum of twenty environmental samples. A preparation batch is composed of environmental sample of the same matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) and/or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices.

BIAS - the deviation of the mean of replicate analytical measurements from a reference analyte concentration. Relative bias is represented by analytical measurement mean minus the reference analyte concentration and the difference divided by the reference analyte concentration. See accuracy and precision.

4-BROMO-FLUOROBENZENE (BFB) - the compound used to check the tuning of the instrument, and is used as a surrogate compound spiked into every sample for volatile analysis. A compound utilized in EPA gas chromatography/mass spectrometry (GC/MS) volatile methods to establish mass spectral instrument performance.

BIOCHEMICAL OXYGEN DEMAND (BOD) - A measure of the amount of oxygen consumed in the biochemical processes that break down organic matter in water. A larger BOD value indicates a greater degree of organic pollution. A related term, BOD<sub>5</sub>, is the amount of dissolved oxygen consumed in five days.

BIOSEED - the bacterial culture used to inoculate a sample for testing.

BLANK - an artificial sample designed to monitor the introduction of artifacts into the measurement process. For aqueous samples, reagent water is used as a blank matrix. There are several types of blanks, which monitor a variety of processes: 1.) *laboratory blank* is taken through sample preparation and analysis only. It is a test for contamination in sample preparation and analyses. 2.) *storage blank* is stored and analyzed with samples at the laboratory. It is a test for contamination in sample storage as well as sample preparation and analysis. 3.) *trip blank* is shipped to and from the field with the sample containers. It is not opened in the field and, therefore, provides a test for contamination from sample preservation, site conditions, and transport as well as sample storage, preparation, and analysis. It is most commonly used for volatile organics. 4.) *field blank* is opened in the field and tests for contamination from the atmosphere as well as those activities listed under the trip blank.

BNA - base, neutral and acid extractable compounds. The term base, neutral and acid refer to the pH condition of the sample undergoing extraction. Certain compounds extract more efficiently from water under acidic conditions. Such compounds are often referred to as "acid extractables."

CALIBRATE - to determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device, or the correct value for each setting of a control knob. The levels of the applied calibration standard should bracket the range of planned or expect sample measurements.

CALIBRATION - The systematic determination of the relationship of the response of the measurement system to the concentration of the analyte of interest.

Instrument calibration performed before any samples are analyzed is called the initial calibration. Subsequent checks on the instrument calibration performed throughout analysis are called continuing calibration verification.

CALIBRATION BLANK (CB) - a volume of reagent water in the same matrix as the calibration standards but without the analyte.

CALIBRATION CURVE - the graphical relationship between the known values, such as concentrations, of a series of calibration standards and their analytical response.

CALIBRATION METHOD - a defined technical procedure for performing a calibration.

CALIBRATION STANDARD - a certified material used to calibrate an instrument.

CERTIFIED REFERENCE MATERIAL (CRM) - reference material, one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30)

CONTINUING CALIBRATION VERIFICATION (CCV) - used to evaluate the performance of the instrument system with respect to a defined set of method criteria. The CCVs are used to verify instrument performance during analysis. The CCV shall be prepared from the same stock standard solution used to prepare the calibration curve.

CHAIN-OF-CUSTODY-Procedures and associated documents designed to trace the custody of a sample from the point of origin to final disposition, with the intent of legally demonstrating that custody remained intact and the tampering or substitutions were precluded.

CHEMICAL OXYGEN DEMAND (COD) - A measure of the oxygen required to oxidize all compounds in water, both organic and inorganic.

COMBINED STANDARD UNCERTAINTY - The standard uncertainty of the analytical measurement result that is the sum in quadrature (square-root-of-the-sum-of-the-squares) of the component standard uncertainties.

CORRECTIVE ACTION - the action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation to prevent reoccurrence.

COVERAGE FACTOR - the numerical factor used as a multiplier of the combined standard uncertainty to expand the uncertainty corresponding to a specific level of confidence. The Student's *t*-distribution is used for determining the coverage factor.

DEMONSTRATION OF CAPABILITY (DOC) - a procedure used to establish the ability of the analyst to generate acceptable accuracy.

DEIONIZED WATER (DI) - water from which the majority of ions have been removed. Water that is contaminant free and used in the laboratory, the resistivity should be  $\leq 18$  mega ohms.

DISSOLVED METALS -- metallic elements determined on a water sample that has been passed through a 0.45-um filter.

DISSOLVED OXYGEN (DO) - the oxygen freely available in water, an indicator of water quality.

DISSOLVED SOLIDS - disintegrated organic and inorganic material contained in water. Excessive amounts make water unfit to drink or use in industrial processes.

DRY WEIGHT - the weight of a sample based on percent solids. The weight of a sample after drying in an oven at a specified temperature.

DUPLICATE MEASUREMENT - a duplicate measurement is a second measurement made on the same or identical sample of material to assist in the evaluation of measurement of variance.

DUPLICATE SAMPLE - two samples taken from the same population and carried through certain stages of sampling and testing. Duplicate sample include field co-located duplicate samples, field-split duplicate samples, and laboratory duplicate subsamples.

EXPANDED UNCERTAINTY - the quantity defining an interval enveloping the analytical measurement that captures a large fraction of the distribution of analyte concentrations that could be attributable to the quantity measured. The combined standard uncertainty is multiplied by the coverage factor to calculate the expanded uncertainty.

EXTERNAL STANDARDS - a method of quantifying chromatographic data in which standards of known concentration are analyzed prior to unknown samples. The chromatographic peak area (or height) of a sample component is compared to a calibration curve of a peak area constructed from the standard data for that component. This comparison allows the concentration of the component in the sample to be determined.

FECAL COLIFORM BACTERIA - bacteria found in mammals' intestinal tracts. Their presence in water or sludge is an indicator of pollution and possible contamination by pathogens.

FLAME IONIZATION DETECTOR (FID) - a gas chromatography detector in which the column effluent gas is mixed with hydrogen and burned in air or oxygen. The ions and electrons produced in the flame generate an electric current proportional to the amount of materials in the detector. The FID responds to nearly all organic compounds, but it does not respond to air and water, which makes it exceptionally suited to environmental analysis.

FLASHPOINT - the lowest temperature at which a flammable liquid gives off sufficient vapor to form an ignitable mixture with air near its surface or within a vessel. Combustion does not continue.

GAS CHROMATOGRAPHY (GC) - a technique for detecting organic compounds by using their physical and chemical properties to separate a mixture. The compounds are identified and quantified with various types of detectors as they exit the chromatograph. Selection of detectors is dependent on the particular compounds of interest.

GAS CHROMATOGRAPHY MASS SPECTROMETRY (GCMS) - a technique in which sample analytes are bombarded with electrons as they exit a gas chromatography column and are fragmented into characteristic ion patterns. The mass spectrometer is the detector. It can determine which fragments are present and therefore the identity of the compounds.

GLUCOSE GLUTAMIC ACID (GGA) - used as a laboratory control standard in BOD procedures.

GRAPHITE FURNACE - a technique used for the analysis of metals. An atomic absorption spectrophotometer heats the sample within a graphite tube using an electrical current. It is also commonly called a flameless furnace and

generally provides greater sensitivity for certain metals than flame or inductively coupled argon plasma techniques.

GRAVIMETRIC - analyses based on the direct or indirect weighing of the analyte in question. This technique usually requires the use of an analytical balance with a sensitivity of 0.1 mg or better.

HALL ELECTROLYTIC CONDUCTIVITY DETECTOR - an element-selective gas chromatography detector primarily intended for trace analysis of organic compounds containing chlorine, nitrogen or sulfur. In operation, this detector pyrolyzes the column effluent gas into soluble electrolytes that are dissolved in a stream of deionized liquid. The observed change in electrical conductivity, proportional to the amount of material present, is measured.

HAZARDOUS WASTE - waste regulated under RCRA that can pose a substantial or potential hazard to human health or the environment when improperly managed. Such wastes possess at least one of four characteristics (ignitability, corrosivity, reactivity, or toxicity) or appear on special EPA hazardous waste lists. The term is not interchangeable with hazardous substances or material.

HEADSPACE - Any area in a container not completely filled by the sample in which gases can collect.

HEAVY METALS - metallic elements with high atomic weights, i.e., mercury, chromium, cadmium, arsenic, and lead. They can damage the health of plants and animals at low concentrations and tend to accumulate in the food chain.

HOLDING TIME - the maximum times that samples may be held before analysis and still be considered valid or not compromised. The storage time allowed between sample collection and sample analysis when the designated preservation and storage techniques are employed.

HYDROCARBONS - chemical compounds that consist entirely of carbon and hydrogen.

ICP - Inductively coupled plasma is a type of instrument used for metals analysis. Because the temperature of the plasma is considerably higher (10,000°K) than the temperature of a flame atomic absorption spectrophotometer, it is especially useful for refractory metals. Some instruments are also capable of performing simultaneous multielement analysis.

ICP-MS - Inductively coupled plasma mass spectrometry (ICP-MS) is a type of mass spectrometry highly sensitive and capable of the determination of a range of metals and several non-metals at concentrations below one part in 10<sup>12</sup> (part per trillion). It is based on coupling together an inductively coupled plasma as a method of producing ions with a mass spectrometer as a method of separating and detecting the ions. In trace elemental analysis, the method has advantages of high speed, precision and sensitivity.

IGNITABLE - capable of burning or causing a fire.

INORGANIC CHEMICALS - chemical substances of mineral origin, unlike organic chemicals whose structure relies on carbon atoms.

INITIAL CALIBRATION VERIFICATION (ICV) - also referred to as the Independent Calibration Verification for this laboratory. A standard solution used to verify the calibration curve derived from a source independent of the instrument calibration standard. The ICV is used to quantify second source standard variance and bias.

INSTRUMENT CALIBRATION STANDARD - a reference material used to standardize an analytical instrument.

INSTRUMENT DETECTION LIMIT (IDL) - a term utilized in the EPA Inorganic Contract Laboratory Program. The IDL is three times the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration of three to five times that of the IDL on three nonconsecutive days with seven consecutive measurements per day. The IDL is the concentration equivalent to a signal, due to the analyte of interest, which is the smallest signal that can be distinguished from background noise by a particular instrument. The IDL should always be below the method detection limit, and is not used for compliance data reporting, but may be used for statistical data analysis and comparing the attributes of different instruments.

INSTRUMENT PERFORMANCE CHECK - The analyses of one of the ICSs to verified initial and continuing calibration (CCV). The IPC is used to quantify the instrumental testing repeatability variance and bias.

INSTRUMENT TUNING - a technique used in gas chromatography/mass spectrometry procedures to verify that the instrument is properly calibrated to produce reliable mass spectral information. See 4-Bromofluorobenzene under "B".

INTERNAL STANDARDS - compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for volatile organics), and sample extract (for semivolatiles) at a known concentration, prior to analysis. Internal standards are used as the basis for quantification of the target compound.

IONIZATION - Utilized in mass spectrometry to fragment analyte molecules into smaller segments. These smaller mass segments are then separated and plotted to form a "mass spectrum" which is used to identify the parent molecule. Electron impact is one example of ionization used in mass spectrometry. In more technical terms. Ionization is the process by which neutral atoms or groups of atoms become electrically charged, either positively or negatively, by the loss or gain of electrons.

ISOMERS - chemical compounds with the same molecular weight and atomic composition by differing molecular structure, e.g., n-pentane and 2-methylbutane.

LABORATORY CONTROL SAMPLE (LCS) - a clean-matrix reference sample spiked with a standard with an established analyte concentration. The LCS is carried through the entire chemical preparation and testing procedures. The LCS is used to quantify the variance and bias of the chemical preparation and instrumental testing stages without matrix interference. Also called a Laboratory fortified blank (LFB).

LABORATORY CONTROL SAMPLE DUPLICATE (LCSD) - a duplicate of the laboratory control sample in a clean matrix that is carried through the entire chemical preparation and testing procedures.

LABORATORY DUPLICATE SAMPLE (LAB DUPLICATE) - a portion of the collected sample that is carried through the chemical preparation and testing. The laboratory duplicate subsample is used to quantify the variance of the chemical preparation and instrumental testing stages with matrix interferences.

LEACHATE - a liquid that results from water collecting contaminants as it trickles through wastes, agricultural pesticides or fertilizers. Leaching may occur in farming areas, feedlots, and landfills, and may result in hazardous substances entering surface water, groundwater or soil.

LDEQ - Louisiana Department of Environmental Quality

LIBRARY SEARCH - a technique in which an unknown mass spectrum of a compound is compared to the mass spectra of compounds contained in a computer library in an

effort to identify the compound. Compounds identified in this manner are referred to as tentatively identified compounds (TICs).

LIMIT OF DETECTION (LOD) - an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent.

LIMIT OF QUANTIFICATION (LOQ) - the minimum levels, concentrations, or quantities of a target variable (e.g. target analyte) that can be reported with a specified degree of confidence..

LIMS - laboratory information management system. Horizons is the LIMS used by GCAL.

LINEAR CALIBRATION RANGE - the concentration range over which the instrument response is linear.

LOG-IN - the receipt and initial management of an environmental sample. It generally includes identifying who sent the sample; maintaining chain-of-custody; checking report and invoice information; recording analysis requested, including methodology and special instructions; and assigning a discreet in-lab identification, usually a number or bar code.

MASS SPECTRUM - a plot of ion mass/charge ratio versus intensity. A fragmentation pattern results from the particles whose mass distribution is characteristic of the parent molecule. Qualitative information is provided by a mass spectrum.

MATERIAL SAFETY DATA SHEET (MSDS) - a compilation of information required under the OSHA Communication Standard on the identity of hazardous chemicals and their associated health and physical hazards, exposure limits and precautions.

MATRIX - The physical characteristics or state of a sample - e.g., water, soil, sludge.

MATRIX INTERFERENCE - the influence of the sample matrix or sample components upon the ability of qualitatively identifies and quantitatively measures compounds in environmental samples.

MATRIX MODIFIERS - chemicals added to samples for metals analysis, which are used to lessen the effects of chemical interference, viscosity and surface tension.

MATRIX SPIKE (MS) - aliquot of a sample fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery of the spike. Matrix spiked samples are used to quantify the variance and bias of the chemical preparation and testing stages with matrix interference.

MATRIX SPIKE DUPLICATE (MSD) - a second aliquot of the same matrix as the matrix spike that is spiked to determine the precision of the method.

METHOD DETECTION LIMIT (MDL) - the minimum concentration of compound that can be measured and reported within 99% confidence that the value is above zero and is determined from analysis of a sample in a given matrix containing the analyte.

MUST - denotes a mandatory requirement.

NARRATIVE - in an analytical report, a descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

NUTRIENT - any substance assimilated by living things that promote growth. The term is generally applied to nitrogen and phosphorus in wastewater, but is also applied to other essential and trace elements.

ORGANIC - generally, any compound that contains carbon bonded to a hydrogen or halogen atom.

OXIDATION - the process in chemistry whereby electrons are removed from a molecule.

PCBs - Polychlorinated biphenyl, a group of toxic persistent chemicals used in transformers and capacitors for insulating purposes and in gas pipeline systems as a lubricant. Sale of PCBs for new uses was banned by law in 1979.

PERCENT RECOVERY - a measure of accuracy that is calculated as the measured value relative to the true value expressed as a percent.

PERFORMANCE AUDIT - a quantitative evaluation of a measurement system that involves the analysis of standard reference samples or materials which are certified as to their chemical composition or physical characteristics.

PROFICIENCY TEST (PT) SAMPLE - a sample of known composition (unknown to the laboratory), provided by an external source, which is used to evaluate lab performance. For certification purposes the PT sample is purchased from an approved provider.

pH - a numerical designation of relative acidity and alkalinity. A pH of 7.0 indicates precise neutrality. Progressively higher values indicate increasing alkalinity and lower values increasing acidity.

POLLUTANT - generally, any substance introduced into the environment that adversely affects the usefulness of a resource.

PRACTICAL QUANTITATION LIMIT (PQL) - the lowest level that can be reliably achieved within specified limits of precision and accuracy routine laboratory operating conditions. The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value.

PRECISION - a measure of the ability to reproduce analytical results. It is generally determined through the analysis of duplicate samples. The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range in either absolute or relative terms.

PRESERVATIVE - a chemical or reagent added to a sample to prevent or slow decomposition or degradation of a target analyte or a physical process. Physical and chemical preservation may be used in tandem to prevent sample deterioration.

PURGE AND TRAP - a technique used in the analysis of volatile organic where analytes are purged from a sample by means of an inert gas and trapped on a sorbent column. The sorbent is then flashheated and the analytes are transferred onto a gas chromatographic column for separation and identification.

PURGEABLE ORGANIC - an organic compound that is generally less than 20% soluble in water and has a boiling point at or below 200°C. A volatile organic. An organic compound is generally considered to be purgeable if it can be removed from water using the purging process.

QC BASED NESTED APPROACH - a procedure for estimating the uncertainty of measurement using quality control data. The procedure is described in SOP QA-013.

QUALITY ASSURANCE (QA) - all those planned and systematic actions necessary to provide adequate confidence in laboratory results. An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets the defined standards of quality with a stated level of confidence.

QUALITY ASSURANCE PROGRAM PLAN - a written assembly of management policies, objectives, principle and general procedures that outlines how the laboratory intends to generate data of known and accepted quality.

QUALITY CONTROL (QC) - the overall system of technical activities whose purpose to measure and control the quality of a product or service so that it meets the needs of the users.

QUALITY CONTROL CHART - a graph of analytical measurement results for a specific QC standard plotted sequentially with upper and lower control limits ( $\pm 3SD$ ). A central line that is the best estimate of the average variable plotted, and upper and lower marginal exceedence limits ( $\pm 4SD$ ) are usually included in the Quality Control Chart. The chart may also include upper and lower warning limits ( $\pm 2SD$ ).

QUALITY SYSTEM - a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items) and services. The quality system provides the framework for planning, implement and assessing work performed by the organization and for carrying out required QA and QC.

$r^2$  - Correlation Coefficient Squared - parameter used to evaluate the fit of linear or quadratic regression curve fit of initial calibration data.

RAW DATA - any original information from a measurement activity or study recorded in a laboratory note book, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfilm copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

REACTIVITY - the tendency of a chemical to explode under normal management conditions, to react violently when mixed with water, or to generate toxic gases.

REAGENT WATER - water in which an interference is not observed at or above the minimum quantitation limit of interest.

REFERENCE MATERIAL - a traceable standard with an established analyte concentration. Material or substance whose property values are sufficiently homogenous, stable and well established to be used for the calibration of an apparatus, the assessment of a measurement method or for assigning values to materials (ISO Guide 30)

REFERENCE METHOD - a method of known and documented accuracy and precision issued by an organization recognized as competent to do so.

RELATIVE RETENTION TIME - a measure of the shift in retention time of an analyte when referenced to an internal standard.



RELATIVE RESPONSE FACTOR (RRF) - a measure of the relative mass spectral response of an analyte compared to its internal standard. RRFs are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

REPLICATE ANALYSES - two or more results representing the same sample parameter. Replicate analyses are used to quantify the analytical measurement repeatability precision.

RPD - Relative Percent Difference, a relative difference between two analyses calculated by dividing the absolute difference of those two results by the average of their two values, then multiplying by 100.

RESOLUTION - the degree of separation between peaks eluting from a chromatographic column. Sufficient resolution between peaks is required for proper quantitation of unknown analytes.

RETENTION TIME - a term used in gas and liquid chromatography describing the time elapsed from the sample injection until the specific compound elutes or exits the chromatographic column at the detector. Each compound has a characteristic retention time on a specific column; therefore, this information is used to qualitatively identify the compounds in the sample.

SAMPLE - portion of material collected for chemical analysis, identified by a unique number assigned by the LIMS.

SHALL - denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled.

SHOULD - denotes a guideline or recommendation whenever noncompliance with the specification is permissible.

SOLID WASTE - non-liquid, non-soluble materials, ranging from municipal garbage to industrial waste, that contain complex, and sometimes hazardous, substances. Solid wastes include sewage sludge, agricultural refuse, demolition wastes, mining residues, and even liquids and gases in containers.

SOLVENT - a substance, usually liquid, capable of dissolving or dispersing one or more other substances.

SPIKE - a known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

STANDARD CURVE - a curve that plots concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by diluting the stock analyte solution in graduated amounts that cover the expected range of the samples being analyzed. The calibration standards must be prepared by using the same type of acid or solvent at the same concentration as for the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

STANDARD OPERATING PROCEDURE - a detailed written description of how a laboratory executes a particular procedure or method intended to standardize its performance.

STANDARDIZED REFERENCE MATERIAL (SRM) - a certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method.

STANDARD UNCERTAINTY - the analytical measurement uncertainty expressed as a standard deviation. The relative standard deviation represents the relative standard uncertainty.

SURROGATE - an organic compound similar to the analyte of interest in chemical composition, extraction and chromatography, but not normally found in environmental samples. Primarily used in chromatography techniques, the surrogate is spiked into quality control blanks, calibration and check standards, samples (including duplicated and QC reference samples) and spiked samples before analysis. A percent recovery is calculated for each surrogate.

SUSPENDED SOLIDS - small pollutant particles that float on the surface of, or are suspended in, sewage or other liquids. They resist removal by conventional means.

TARGET COMPOUND - specific compounds that are to be quantified in a sample based on a standard list of potential compounds.

TENTATIVELY IDENTIFIED COMPOUNDS (TICs) - compounds detected in samples that are not target compounds, internal standards, system monitoring compounds or surrogates. TICs usually consist of up to 30 peaks that are greater than 10% of the peak areas, or heights, of the nearest internal standard. They are subjected to mass spectral library searches for tentative identification. A client may specify the number of unknown peaks in its samples it wishes the laboratory to tentatively identify.

TOTAL METALS - metallic elements that have been digested prior to analysis.

TYPE A EVALUATION UNCERTAINTY - the method of evaluation of uncertainty by the statistical analysis of a series of test results.

TYPE B EVALUATION OF UNCERTAINTY - the method of evaluation of uncertainty by means other than statistical analysis.

UNCERTAINTY - the parameter associated with the analytical measurement results that characterizes the dispersion of the values that could be reasonable attributed to the quantity measured.

UNCERTAINTY INTERVAL - the range of analyte concentrations that an analytical measurement could represent at a specified level of confidence. The relative standard deviation is used to represent the relative standard uncertainty in the QC-based Nested Approach.

VALIDATION - the process for evaluating the completeness, correctness, and conformance/compliance of a specific data set against the method, procedural, or contractual specifications. It essentially evaluates performance against predetermined specifications.

VOA - volatile organic analysis

VOA BOTTLE - a vial used to contain samples for volatile organic analysis.

VOLATILE COMPOUNDS - compounds amenable to analysis by purge and trap. Synonymous with purgeable compounds.

VOLATILE ORGANIC COMPOUND (VOC) - any organic compound that participates in atmospheric photochemical reactions, except for those designated by the EPA Administrator as having negligible photochemical reactivity.

WET CHEMISTRY - procedures that involve distillations, colorimetric determinations and titrimetric measurements. Examples are chloride, nitrates, sulfates and biochemical oxygen demand.

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STANDARD OPERATING PROCEDURE FOR  
3050 METALS DIGESTION

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REVISION NO. 14

GULF COAST ANALYTICAL LABORATORIES, INC.  
METALS  
STANDARD OPERATING PROCEDURE

PROCEDURES: MET-004  
PAGE: 1 OF 6  
EFFECTIVE DATE: 03/28/11  
APPROVED BY:  
QA/QC APPROVED:

SUBJECT

SCOPE AND APPLICATION

This digestion procedure is designed for the preparation of sediment, sludge and soil samples for determination of total metals by furnace atomic absorption or by ICP.

MATRIX

Solid

REFERENCE

SW846 Method 3050B

SAMPLE COLLECTION

Samples may be collected and stored in plastic or glass containers.

HOLDING TIME

6 months

DEFINITIONS

See SOP GEN-016

SAFETY

Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards, and all associated protective wear and spill clean-up procedures PRIOR TO the use of any chemical. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents and chemicals must be handled in ventilated hoods.

APPARATUS &  
REAGENTS

70 mL digestion vessels  
250 mL glass digestion flasks  
Watch glasses - ribbed  
100 mL graduated cylinders  
Specimen container  
Nitric Acid -- Trace Metal grade  
Hydrochloric Acid -- Trace Metal grade  
30% Hydrogen Peroxide  
DI grade water  
CPI Mod Block - Digestion Block

Hot plate  
Balance accurate to 0.01g  
Teflon boiling beads (or equivalent)

STANDARDS

GCAL 1 MIX - 4000 mg/L Na; 2000 mg/L K; 1000 mg/L Al, Ca, Fe, Mg; 100 mg/L Ag, As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, P, Pb, Se, Sr, Tl, V, Zn

GCAL 2 MIX - 500 mg/L b; 100 mg/L Mo, Sb, Sn,

GFAA spiking solution - 4.0 mg/L As, Se, Tl prepared by the metals laboratory

ICP-MS spike - 100mg/L Na, Ca, Fe, Mg, K; 20mg/L Al, Zn; 10mg/L trace.

PROCEDURE

1. Homogenize the wet sample by mixing well. The sample may be removed from the container and placed on butcher paper to allow for mixture of the whole sample. Weigh  $1.25\text{g} \pm 0.1\text{g}$  of a homogenized sample into a labeled 70 mL digestion vessel. A minimum weight may be required to meet the project required detection limit. Check with the metals supervisor or project manager to determine if a minimum weight is required. Record the weight to the nearest 0.01g. Prepare QC samples as required in the quality control section.
2. Add 5 mL of 1:1  $\text{HNO}_3$  and mix.
3. Set Mod Block to the required setting for temperature of 90-95°C. Allow 15 minutes for the block to stabilize at the appropriate temperature.
4. Place the digestion vessels in the block, cover with a watch glass and reflux the samples for 15 minutes without boiling.
5. Cool and add 2.5 mL of concentrated  $\text{HNO}_3$  to each sample, replace the watch glasses and reflux for 30 minutes.
6. If brown fumes are generated, indicating oxidation of the sample by the  $\text{HNO}_3$ , repeat step 5 over and over until no brown fumes are given off by the sample indicating the reaction is complete.
7. Cool. Add 1 mL DI  $\text{H}_2\text{O}$  and 1.5 mL 30% of  $\text{H}_2\text{O}_2$  (peroxide) and cover with the watch glass.

8. On low heat, warm the sample to start the  $\text{H}_2\text{O}_2$  reaction and heat until effervescence subsides. Continue adding 30%  $\text{H}_2\text{O}_2$  in 1 mL aliquots (up to a maximum total of 5 mL of 30%  $\text{H}_2\text{O}_2$ ) until the reaction is minimal.
9. Cover the sample with the watch glass and heat at  $95^\circ\text{C}$  for 2 hours or until the sample volume is about 5mL.
10. For ICP and ICP-MS preparation only, add 5 mL of concentrated HCl, cover with a watch glass and reflux at  $95^\circ\text{C}$  for 15 minutes.
11. Cool and adjust final volume to 50 mL with DI  $\text{H}_2\text{O}$ .
12. Document digestion by completing prep sheet (attached).
13. Filter through Whatman 41 (or this can be done in the lab) prior to analysis.

Most liquid organic samples are digested on a hot plate due to the extended digestion time.

#### HOT PLATE PROCEDURE

1. Homogenize the sample by mixing well. Mix in a well-ventilated area. Weigh  $1.25\text{g} \pm 0.1\text{g}$  of a homogenized sample into a labeled 250 mL glass digestion vessel. A minimum weight may be required to meet the project required detection limit. Check with the metals supervisor or project manager to determine if a minimum weight is required. Record the weight to the nearest 0.01g. Prepare QC samples as required in the quality control section.
2. Add 5 mL of  $\text{HNO}_3$  and mix.
3. Set hot plate to the required setting for temperature of  $90\text{--}95^\circ\text{C}$ . Allow 15 minutes for the hot plate to stabilize at the appropriate temperature.
4. Place the digestion flasks on the hot plate, cover with a watch glass and reflux the samples for 15 minutes without boiling.
5. Cool and add 2.5 mL of concentrated  $\text{HNO}_3$  to each sample, replace the watch glasses and reflux for 30 minutes.

6. If brown fumes are generated, indicating oxidation of the sample by the  $\text{HNO}_3$ , repeat step 5 over and over until no brown fumes are given off by the sample indicating the reaction is complete. For concentrated organic samples, this step is performed many times and therefore the digestion may take several days.
7. Cover with watch glass. Heat at  $95^\circ\text{C}$  for 2 hours or until the sample volume is about 5 mL.
8. Cool. Add 1 mL DI  $\text{H}_2\text{O}$  and 1.5 mL 30% of  $\text{H}_2\text{O}_2$  (peroxide) and cover with the watch glass.
9. On low heat, warm the sample to start the  $\text{H}_2\text{O}_2$  reaction and heat until effervescence subsides. Continue adding 30%  $\text{H}_2\text{O}_2$  in 1 mL aliquots (up to a maximum total of 5 mL of 30%  $\text{H}_2\text{O}_2$ ) until the reaction is minimal.
10. Cover the sample with the watch glass and heat at  $95^\circ\text{C}$  for 2 hours or until the sample volume is about 5mL.
11. For ICP and ICP-MS preparation only, add 5 mL of concentrated  $\text{HCl}$ , cover with a watch glass and reflux at  $95^\circ\text{C}$  for 15 minutes.
12. Filter through Whatman 41 (or this can be done in the lab).
13. Cool and adjust final volume to 50 mL with DI  $\text{H}_2\text{O}$ .
14. Document digestion by completing prep sheet (attached).

OPTIONAL PROCEDURE  
FOR ANTIMONY (Solid)

The following procedure may be used to improve the solubility and recovery of Antimony when necessary. These steps are optional and are not required on a routine basis.

1. Weigh 1.25g of sample in digestion tube (same as original prep).
2. Add 1.5mL concentrated  $\text{HNO}_3$  and 5 mL concentrated  $\text{HCl}$ .
3. Set Mod Block to the required setting for temperature of  $95^\circ\text{C} \pm 5^\circ\text{C}$ . Allow 15 minutes for the block to stabilize at the appropriate temperature.

4. Place the digestion vessel in the block, cover with watch glass and reflux for 15 minutes.
5. Filter the digestate through Whatman #41 filter paper (or equivalent) and collect filtrate in another labeled 70mL digestion vessel. While still in funnel, rinse the filter paper with no more than 2.5 mL of hot HCl (~95 °C), then with 10mL of hot (~95 °C) reagent water. Collect washings in same digestion vessel as the filtrate.
6. Remove filter and residue from funnel and place in original digestion vessel. Add 2.5 mL concentrated HCl, place vessel back in block with watch glass and sides of digestion vessel with reagent water. Filter the residue into the sample digestion vessel as above.
7. Allow sample to cool\* and bring to volume, 50 mL.

\* If precipitation occurs in digestion tube upon cooling of the primary or secondary filtrate do not bring up to volume. Instead, add up to 5mL of concentrated HCl to dissolve precipitate. Once precipitate is dissolved, bring to volume (50 mL) with reagent water.

QUALITY CONTROL    ICP and ICP-MS

With each batch of samples, the following will be digested:

- a. Prepare one method blank (MB)(approximately 1.25g of boiling beads, document exact weight) with each batch of twenty or fewer samples.
- b. Prepare one Laboratory Control Sample (LCS) (approximately 1.25g of boiling beads, document exact weight) with each batch of twenty or fewer samples. Add 0.25 mL of GCAL-1 & GCAL-2 for ICP-AES. (Check to be sure all target elements are contained in spike solutions. If not, add them separately from stock solutions). For ICP-MS add 0.25mL of ICP-MS spike.
- c. Duplicate one sample with each batch of twenty or fewer samples.
- d. Prepare one matrix spike sample (MS) with each batch of twenty or fewer samples. Add 0.25 mL of GCAL-1 & GCAL-2 for ICP-AES. (Check to be sure all target elements are contained in spike solutions. If not, add them separately from stock solutions). Matrix spike duplicates may be performed if required for a

specific project. For ICP-MS add 0.25mL of ICP-MS spike.

- e. Record the laboratory ID for all standards and reagents in the prep sheet.

#### GRAPHITE FURNACE

With each batch of samples, the following will be digested:

- a. Prepare one method blank (MB)(approximately 1.25g of boiling beads, document exact weight) with each batch of twenty or fewer samples.
- b. Prepare one Laboratory Control Sample (LCS) (approximately 1.25g of boiling beads, document exact weight) with each batch of twenty or fewer samples. Add 0.5 mL of GFAA spiking solution.
- c. Duplicate one sample with each batch of twenty or fewer samples.
- d. Prepare one matrix spike sample (MS) with each batch of twenty or fewer samples. Add 0.5 mL GFAA spiking solution. Matrix spike duplicates may be performed if required for a specific project.
- e. Record the laboratory ID for all standards and reagents in the prep sheet.

#### METHOD PERFORMANCE

In a single laboratory, the recoveries of the three matrices presented in Table 2 of Method 3050 were obtained using the digestion procedure outlined for samples prior to analysis by FLAA and ICP-AES. The spiked samples were analyzed in duplicate. Tables 3-5 of Method 3050 represents results of analysis of NIST Standard Reference Materials that were obtained using both atmospheric pressure microwave digestion techniques and hot-plate digestion procedures.

POLLUTION PREVENTION See QAPP Section 10.2

WASTE MANAGEMENT See SOP GEN-009



GULF COAST ANALYTICAL LABORATORIES, INC.  
METALS  
STANDARD OPERATING PROCEDUREPROCEDURE: MET-005  
PAGE: 1 OF 4  
EFFECTIVE DATE: 03/28/11  
APPROVED BY:  
QA/QC APPROVED:

## SUBJECT SCOPE AND APPLICATION

This digestion procedure is designed for the preparation of aqueous samples, TCLP extracts and EPTOX extracts. This is a vigorous digestion with nitric and hydrochloric acid for the determination of total metals by inductively coupled plasma spectroscopy (ICP).

MATRIX Water

REFERENCES SW846 3010A

PRESERVATION Dissolved Metals -- Filtered through a 0.45 µm membrane filter then HNO<sub>3</sub> to pH <2, analyst must wait at least 24 hours before digesting the sample  
Total Metals -- HNO<sub>3</sub> to pH <2

SAMPLE COLLECTION Samples may be collected and stored in plastic or glass containers.

HOLDING TIMES Dissolved Metals - 6 months  
Total Metals - 6 months

DEFINITIONS See SOP GEN-016

SAFETY Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO the use of any chemical. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents must be handled in ventilated hoods.

APPARATUS 250 mL beakers  
Ribbed and plain watch glasses  
50 mL graduated cylinders  
Specimen containers  
Hot Plate

CPI Mod Block - Digestion Block  
70 mL Digestion Tubes  
Mechanical pipet and clear plastic pipet tips  
Wide range pH paper

REAGENTS  
AND STANDARDS

Label all containers and squeeze bottles with reagent ID, lot, and expiration date.  
All standards used are pure material or from prepared certified solutions. The certificate of analysis shall be kept on file. Follow manufacturer's instruction for standard expiration and storage. Label all working standards using completed standard labels.  
Nitric Acid -- Trace Metal grade  
HCl -- Trace Metal grade  
Spiking Solution for samples and LCS  
    GCAL-1: certified custom solution  
    GCAL-2: certified custom solution  
    ICP-MS spike: certified custom solution  
Deionized water

PROCEDURE

BLOCK DIGESTION SYSTEM

1. Record all sample information in the metals preparation form (attached).
2. Measure 50 mL of a homogenized sample in a 70 mL digestion tube labeled with sample number, date, and prep for all TCLP liquid and aqueous samples. For TCLP organic liquids measure 5mL of sample. Add 1.5 mL concentrated HNO<sub>3</sub> and cover with ribbed watch glass or equivalent.
3. Place tubes in the digestion block. Select Block A or B. Set temperature to the required setting for a temperature of 90-95°C. "B" key for up and "E" key for down. When the desired temperature is reached; Press Enter (C key): 2 hours will show for timer; Press Enter (C key): start time will show; Press Enter (C key): Block will heat to 95°C and maintain heat for 2 hours. Place a thermometer in an uncovered digestion tube to monitor temperature. Record the temperature in the MOD Block Temperature Logbook.
4. Evaporate to about 5 mL without boiling. (Heat may be increased to get a gentle reflux.)

5. Allow sample to cool. Add 1.5 mL of concentrated  $\text{HNO}_3$ .
6. Reflux and add additional  $\text{HNO}_3$  until the reaction is complete. Reaction is completed when it is odorless and clear.
7. Evaporate to small volume, cool and add 5 mL of 1:1 HCl (1mL 1:1 HCl per 10mL of final solution).
8. Reflux 15 minutes.
9. Cool and adjust final volume to 50 mL with DI  $\text{H}_2\text{O}$ .
10. The sample is now ready for analysis. Deliver to the metals laboratory with a copy of the prep log.

#### QUALITY CONTROL

With each batch twenty or fewer samples, the following will be digested:

- a. Method Blank (BLK) - DI + reagents
- b. Laboratory Control Sample (LCS) - DI + reagents - add 0.25 mL of GCAL-1 and 0.25 mL of GCAL-2 for analysis by ICP-AES. (Check to be sure all elements requested to be run are contained in the spiking solutions. If not, add them separately from stock solutions.) For ICP-MS add 0.25 mL of ICP-MS standard.
- c. Method Blank (extracted blank for TCLP) - TCLP extracts only
- d. Duplicate sample
- e. Matrix Spike - add 0.25 mL of GCAL-1 and 0.25 mL of GCAL-2 for analysis by ICP-AES. (Check to be sure all requested elements are contained in the spiking solutions. If not, add them separately from stock solutions.) For ICP-MS add 0.25 mL of ICP-MS standard.
- f. Matrix Spike Duplicate (performed only if required for a project or requested by the client) - add 0.25 mL of GCAL-1 and 0.25 mL of GCAL-2 for

analysis by ICP-AES. (Check to be sure all requested elements are contained in the spike solutions. If not, add them separately from stock solutions.) For ICP-MS add 0.25 mL of ICP-MS standard.

- g. Record the laboratory ID for all standards and reagents in the logbook.
- h. Specific projects may require additional or specific QC, and must be followed for applicable samples. See Sop GEN-019.

METHOD PERFORMANCE      3010A  
No Data Provided

POLLUTION PREVENTION      See QAPP Section 10.2

WASTE MANAGEMENT      See SOP GEN-009

STANDARD OPERATING PROCEDURE FOR ANALYSIS OF SAMPLES BY ICP-MS	REVISION NO. 2
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GULF COAST ANALYTICAL LABORATORIES, INC  
METALS STANDARD OPERATING PROCEDURE

PROCEDURE: MET-021  
PAGE: 1 of 15  
APPROVAL DATE: July 11, 2011  
EFFECTIVE DATE: July 15, 2011  
APPROVED BY:  
QA/QC APPROVED:

SUBJECT SCOPE AND APPLICATION

This method covers the operation of the Agilent 7700 ICP-MS for analysis of low-level concentrations of metals in water samples, waste extracts, or digests. The procedure is applicable for the analysis of aqueous samples, soils, solids, and domestic or industrial wastes for Total metals, after preparation by the appropriate EPA or SW846, procedure. Dissolved metals, filtered and acid preserved prior to analysis may be analyzed without digestion. Elements currently analyzed using these methods are listed in Table I.

MATRIX Water and Solid

REFERENCES EPA 200.8 - Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma - Mass Spectrometry, Revision 5.4 (EMMC Version)

SW846 6020A  
Agilent Instrument Manuals  
Software Guide

SAMPLE COLLECTION Samples may be collected and stored in plastic or glass containers.

HOLDING TIME & PRESERVATION Dissolved Metals (waters) - Filtered through a 0.45 um membrane filter then HNO<sub>3</sub> to pH < 2; for 200.8 adjust the pH to less than 2 with HNO<sub>3</sub> then hold for a minimum of 16 hours.

Total Metals (waters) - HNO<sub>3</sub> to pH < 2; for 200.8 adjust the pH to less than 2 with HNO<sub>3</sub> then hold for a minimum of 16 hours. Digestion required (SOP MET-005)-180 calendar days; and

Total Metals (solids) - Cool 4°C; Digestion required (SOP MET-004, MET-018)-180 calendar days.

DEFINITIONS See SOP GEN-016

## SAFETY

Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO the use of any chemical. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents must be handled in ventilated hoods.

## INTERFERENCES

Several interferences are possible. These include:

1. Isobaric elemental interference caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and cannot be resolved or corrected for by the mass spectrometer in use. These interferences are addressed by the instrument operating conditions, correction equations, and careful selection and monitoring of mass-to-charge ratios. In general, all direct isobaric interferences are avoided by choosing an interference-free isotope. In some cases this may result in a loss in sensitivity.
2. Physical Interferences are generally considered to be effects associated with the sample nebulization and transport processes. Properties such as change in viscosity and surface tension can cause significant inaccuracies especially in samples that may contain high dissolved solids or acid concentrations. If these types of interferences are present, they must be reduced by diluting the sample. Dissolved solids greater than 0.2% w/v causes interferences and clogging of the pump. Internal standards are used to identify this type of interference.
3. Isobaric polyatomic ion interferences are caused by ions consisting of more than one atom with the same nominal charge-to-mass ratio of the ions of interest. Proper tuning of the plasma and torch conditions to reduce the formation of oxides reduces this interference. In addition the use of a collision cell reduces or eliminates this interference in most matrices.
4. Signals from relatively abundant isotopes can coalesce at the wings of relatively less abundant isotopes leading to loss of resolution and poor quantitation.

5. Memory interferences of isotopes from previous sample runs.

#### APPARATUS

Agilent 7700 ICP-MS  
Agilent ASX-500 Autosampler  
Edwards Vacuum Pump Model E2M18  
Peristaltic pump and tubing  
Autosampler tubes  
Mechanical pipets and pipet tips  
Volumetric Flasks - Class A  
Graduated centrifuge tubes  
Eppendorfs  
Argon and Helium supply with pressure controls  
Compressed air

#### REAGENTS AND STANDARDS

1. Deionized water - monitored each day of use for resistivity. Must be 10MΩ-cm or greater.
2. Concentrated Nitric acid - Trace metals grade, label with date opened and enter into reagent logbook
3. Concentrated hydrochloric acid - Trace metals grade, label with date opened and enter into reagent logbook
4. 5% Nitric Acid/2% Hydrochloric Acid - prepared from the Trace metals grade concentrate
5. 2% Nitric Acid/0.5% Hydrochloric Acid - Calibration Blank
6. Stock standard metals solutions (1000 mg/L) - commercially prepared, label with date opened.
7. Custom mixed standards - commercially prepared, label with date opened.
8. Calibration standards: A custom made solution for calibrations standards is used. See Table I for the elements included and concentration. Prepare all standards in the 2% Nitric Acid/0.5% Hydrochloric Acid.
9. Continuing Calibration Verification (CCV) standard - is prepared using the calibration standard. The CCV includes equal aliquots of the calibration standard and 2% Nitric Acid/0.5% Hydrochloric Acid.
10. Second Source Stock Standards - Two, multi-element custom solutions-commercially prepared.

11. Initial Calibration Verification (ICV) standard and QCS standard - Dilute the appropriate amount of the second source multi-element stock solution to volume with 2% Nitric Acid/0.5% Hydrochloric Acid. The concentration of the elements in the stock solutions vary in concentration, therefore the analyst should prepare this solution such that the majority of the elements are in the middle of the calibration range. This solution must be prepared at least quarterly or more frequently if precipitation is noted. Trace metals must be spiked at  $\leq 100\mu\text{g/L}$ . The QCS is prepped in the same manner of samples and is used for the demonstration of capability.
12. Interference solution stock standards - available commercially.
13. Interferent Calibration Solution A (ICSA) - Pipet 50 $\mu\text{L}$  of the interferent stock Agilent ICS-A into a 50mL graduated centrifuge tube and dilute to volume with 2% Nitric Acid/0.5% Hydrochloric Acid. Final concentrations are: Ca-3000 $\mu\text{g/L}$ , Fe and Na-2500 $\mu\text{g/L}$ , Mg, Al, and K-1000 $\mu\text{g/L}$ , and Ti and Mo-20 $\mu\text{g/L}$ .
14. Interferent Calibration Solution B (ICSAB) - Pipet 50 $\mu\text{L}$  of the interferent stock Agilent ICS-A, and 50 $\mu\text{L}$  of interferent stock Agilent ICS-B into a 50mL graduated centrifuge tube and dilute to volume with 2% Nitric Acid/0.5% Hydrochloric Acid. Final concentrations of interferents are as in (13) above; concentrations of minor elements are 20, 10, or 5 $\mu\text{g/L}$ . Check solution frequently for signs of precipitation and take corrective action when noted.
15. Internal Standard - 1 $\mu\text{g/mL}$  of  $^{45}\text{Sc}$ ,  $^{103}\text{Rh}$ ,  $^{115}\text{In}$ ,  $^{159}\text{Tb}$ ,  $^{175}\text{Lu}$ , and  $^{209}\text{Bi}$
16. Low-level Calibration Verification Standard analyzed at 0.5 $\mu\text{g/L}$  of trace elements.
17. All solutions are prepared in labeled, graduated centrifuge tube.
18. All reagents used must be entered in the reagent logbook, except for water, which is monitored by the QA/QC Department. Standards prepped are entered into the standard prep logbook. See SOP GEN-006 for additional information and documentation requirements.



19. Argon >99.99% purity

20. Helium

#### INSTRUMENT SET-UP

1. Check Argon, Helium, and compressed air supply.
2. Check wash solution in autosampler flush container. Fill if necessary (2% Nitric Acid, 0.5% Hydrochloric Acid).
3. Check drain bottle below instrument. Empty if necessary.
4. Connect the peristaltic pump tubing between the posts on the pump (Sample pump and internal standard pump). Close the platens.
5. Start the computer. Select the ICP-MS Top icon on the Windows Desktop.
6. Select the Plasma ICON. Select "ON" button. Allow Plasma to warm up 45 minutes.
7. Set up sequence by filling in the positions with the samples to be analyzed. Include the standard identification numbers for the tune, calibration standards, ICV, ICSA, and ICSAB on the bench sheet (Attachment 1). Print sequence.
8. Load the autosampler by filling the tubes (10 mL will suffice) and inserting them into the positions corresponding to those on the bench sheet. Verify all blank, standard and QC sample tubes are filled.
9. Save sequence using the date as YYMMDD.
10. Select Load and Run sequence in the ICP-MS window.

#### CALIBRATION AND SAMPLE ANALYSIS

1. The instrument must first pass the mass calibration, resolution, and stability checks or tune check. The mass calibration cannot drift by more than 0.1 amu from the true value. The resolution must be verified to be less than 0.9 amu full width at 10% peak height. A minimum of five replicates are acquired with an RSD of  $\leq 5\%$ . If these criteria are not achieved, perform instrument maintenance and/or tuning. Calibration and sample

analysis cannot begin until acceptable mass calibration and resolution are demonstrated.

2. After verifying that tune passes, continue with initial calibration. Instrument will calibrate and continue with QC and sample analysis. The calibration is a multi-point calibration (minimum of three points) with a blank. It is verified with an ICSA, ICSAB, ICV, LLICV, CCV, LLCCV and CCB. Once data is collected, and instrument QC is evaluated, the data can be posted. This is performed by direct upload of data to the intranet ICP-MS uploader. In ICP Data Analysis window and select tools. Upload selected samples as a csv file.
3. Document all runs on a batch log using the ICP file maker program available on the metals workstation. On each batch sheet document the analyst, date, time, standard identification, dilutions, and reviews. An example sheet is attached.
4. Go to the intranet ICPMS uploader and browse for specific CSV file. Enter the HBN and select import. Select samples to be posted and select export.
5. SHUT DOWN - If an additional run will not be performed add a TERM block and an ERRTERM block at the end of the sequence.
6. Samples are diluted if the detected concentration is  $\geq 90\%$  of the high-level standard as described in QC section of this SOP for E200.8 or above the high-level standard for samples analyzed by SW6020A.
7. Three replicates are determined for all sample, calibration, and QC analysis. If the RSD of the replicates is more than 5%, each replicate is evaluated. Note that an RSD of  $>5\%$  may be typical for low concentrations and data should not be rejected based solely on the RSD. The average of the three replicates is reported.
8. Rinse for a minimum of one minute between sample analyses.

#### REPORTING LIMITS

The following charts indicate typical reporting limits for EPA 200.8, and SW-846 6020A. Lower limits may be achievable. Limits may also be adjusted higher if they meet the client's requirements due to LLCCV or LOQ performance. Project specific limits will be handled in accordance with SOP GEN-019.

**EPA 200.8:**

ANALYTE	WATER (ug/L)
Aluminum	10
Antimony	0.5
Arsenic	0.5
Barium	0.5
Beryllium	0.5
Cadmium	0.5
Chromium	0.5
Cobalt	0.5
Copper	0.5
Lead	0.5
Manganese	0.5
Molybdenum	0.5
Nickel	0.5
Selenium	1.0
Silver	0.5
Thallium	0.5
Vanadium	0.5
Zinc	10.0

**SW-846, 6020A:**

ANALYTE	WATER (ug/L)	SOLID (ug/kg)
Aluminum	10	400
Antimony	0.5	20
Arsenic	0.5	20
Barium	0.5	20
Beryllium	0.5	20
Cadmium	0.5	20
Calcium	50	2000
Chromium	0.5	20
Cobalt	0.5	20
Copper	0.5	20
Iron	50	2000
Lead	0.5	20
Magnesium	50	2000
Manganese	0.5	20
Molybdenum	0.5	20
Nickel	0.5	20
Potassium	50	2000
Selenium	1	40
Silver	0.5	20
Sodium	50	2000
Strontium	0.5	20

Thallium	0.5	20
Tin	0.5	NA
Titanium	0.5	20
Vanadium	0.5	20
Zinc	10	400
Zirconium	0.5	20

QUALITY CONTROL &  
DATA ACCEPTANCE CRITERIA

1. The initial calibration consists of the initial calibration blank and a minimum of three contiguous non-zero points. The lowest non-zero point for each analyte must be at or less than the LOQ. To be considered acceptable the correlation coefficient (r) must be  $\geq 0.998$  for each analyte. Additional points may be analyzed to better define the linear range and the lower limit of quantitation. In addition the high or low points may be removed from a multi-point curve as long as a minimum of three points remain and the reporting limit is adjusted if the low point is removed. Silver is not calibrated above 100ug/L.
2. The Initial Calibration Verification (ICV) standard recoveries must be 90-110%. This is performed daily following the initial calibration. The ICV standard should be prepared from an independent (second source) material at or near the mid-range of the calibration curve.
3. The Initial Calibration Blank (ICB) is used to verify all concentrations are below the client defined reporting limit. The concentration must be  $\leq \pm$  the reporting limit. For DOD projects, concentrations must be less than the LOD. Additional requirements may apply and may be more stringent. See SOP GEN-019. The internal standard response from the ICB is used to calculate drift for all samples in the analytical batch. An ICB cannot be re-analyzed after the calibration to "re-set" the internal standard responses. If for any reason the ICB must be reanalyzed, it must be followed by an ICAL.
4. Analyze a Low-level Initial Calibration Verification (LLICV) prepared at or below the project required reporting limit. The recovery for this check

standard must be 70-130% for 6020A. Recovery limits for 6020A for DOD projects must be 80-120% as established by Table F-8 of the QSM. Project specific limits may be required, see SOP GEN-019.

5. The ICSA solution is used to monitor correction factors for the common interferences. This solution will be used to evaluate elements that are not spiked in the ICSAB. The concentration for these elements must be  $\leq \pm$  the reporting limit. The ICSA concentration must be less than or equal the reporting limit and greater than or equal to -2X the reporting limit. The ICSA is analyzed before sample analysis or every 12 hours, whichever is more frequent. Additional requirements may apply and may be more stringent. See SOP GEN-019.
6. All elements in the ICSAB solution should have recoveries of 80-120%. Results falling outside these limits may indicate the need for re-evaluation of correction equations. The ICSAB is analyzed before sample analysis or every 12 hours, whichever is more frequent.
7. Continuing Calibration Verification standards (CCV), Continuing Calibration Blanks (CCB), and for 6020A Low-level Continuing Calibration Verification (LLCCV) are run every 10 samples and at the end of the analytical run. The CCV recovery must be 90-110%. For 6020A, the recovery for the LLCCV must be 70-130% for all analytes. For DOD projects using 6020A, the recovery for the LLCV must be 80-120% for all analytes as required by Table F-8 of the QSM. The RSD between the replicates should not be greater than 5%. If this is exceeded evaluate the replicates to determine if re-analysis is necessary. The CCB concentration should never exceed the reporting limit. If a CCV, LLCCV, and/or CCB is outside control limits, re-analyze one time (additional runs are acceptable if a documented reason for the failure is available) and if the values are still outside the control limits, recalibrate and re-run the previous 10 samples. Results are reportable with a narrative if the CCB fails and there are no hits in the associated samples. Additional requirements may apply and may be more stringent. See SOP GEN-019.
8. Method Blank - one Method Blank is prepped with each batch of 20 or fewer samples. The concentration of each element should never exceed the reporting limit for all associated samples. Detected levels of

elements in the method blank must be less than 10% of the regulatory limit or the sample concentrations for the data to be reported. For DOD projects, the method blank should be less than ½ the reporting limit. If the data is reported make a note of the problem in the case narrative. Find the source of the problem and correct and re-analyze if possible. Additional requirements may apply and may be more stringent. See SOP GEN-019.

9. A Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD-if required for project or if insufficient sample is available for a matrix duplicate or MS/MSD) is prepped with each batch of 20 or fewer samples. The recovery must be 85-115% for 200.8 and 80-120% for 6020A. The LCS/LCSD RPD should be  $\leq 20\%$ . If the LCS recovery is outside the control limits, the sample batch must be re-prepped. Samples may be reported if the LCS fails high with no hits in associated samples. Report with a narrative describing the failure.
10. A low-level quality control sample (LOQ) is prepped at the same level as the LLICV with initial set-up and quarterly. The sample recovery must be within 70-130% of the true value. Recovery limits for DOD projects must be 80-120% as established by Table F-8 of the QSM. If this sample does not fall within these criteria, evaluate the reason for the failure, perform recalibration and maintenance as necessary and re-prepare the sample. If the sample continues to fall outside of acceptance criteria it may be necessary to increase the reporting limit to a level that falls within this criteria.
11. The Relative Percent Difference (RPD) for matrix duplicates must be  $\leq 20\%$ , unless the sample concentration is less than five times the detection limit or the sample matrix is determined to be heterogeneous. Additional requirements may apply and may be more stringent. See SOP GEN-019.
12. Matrix Spike (MS) and Matrix Spike Duplicate (MSD) recoveries must be 75 - 125% for 6020A water and solids, 70-130% for 200.8. The MS/MSD RPD should be  $\leq 20\%$ . For DOD projects, the MS control limits are the same as the applicable LCS limits. MSD's are prepped and analyzed if there is sufficient sample. If the recovery is outside these limits, the data should be flagged. A spike is not applicable if the spike amount is less than 30% of the concentration

in the native sample. MS/MSD is performed at a minimum of 10% (1 in 10) of samples.

13. For solid samples that require Antimony analysis - If the MS and MSD recovery for Antimony are outside of the control limits of 75-125%, the data should be flagged. In addition, if the recovery for Antimony is <10%, the samples should be reprepared for Antimony only, using the optional procedure for Antimony found in SOP MET-004. Also, if historical data for a particular sample indicates low recoveries for Antimony, the analyst should proceed directly to the optional steps.
14. Interference checks to be performed for 6020A if the MS/MSD fails for any target compound or if requested by the client:
  - A. Post-Digestion Spike -A post-digestion spike is performed on all samples with MS/MSD recoveries outside control limits. A spike is added to a portion of digested sample or its dilution and should be recovered at 80-120% of the known value. For DOD projects the spike should be recovered at 75-125% of the known value. If the recovery is outside control limits perform the dilution test.
  - B. Dilution Test - Perform a 5x dilution on one sample per batch. The concentrations of the elements in the sample to be diluted should be greater than 50 times the MDL. The neat sample concentration and five times the diluted sample concentration should agree within + 10%. If not, a chemical or physical interference should be suspected.
15. Samples with interferent or analyte concentrations  $\geq$  90% of the high level ICAL standard will be diluted and reanalyzed for samples reported by E200.8. Samples reported by SW-846 Method 6020A shall be diluted for any compound greater than the high level calibration standard. Dilute so that the expected concentration is approximately in the mid-level of the curve. Report multiple dilutions if requested by the client.
16. Review the internal standard drift as compared to the calibration blank. The absolute value of any one internal standard must not deviate by more than 70-120% of the original response in the calibration blank. Determine if high or low recoveries are due to instrument drift by reviewing the internal standard recoveries for CCV's and CCB's. This will

eliminate performing unnecessary dilutions if the sample is reanalyzed. High recoveries may be the result of a clogged sample uptake tube or an empty sample vial. Extremely high recoveries can result from the presence of the internal standard element in the original sample. If this is suspected, re-analyze using a different IS. If the analyst determines the recovery is outside the control limits due to matrix interference, then the sample must be reanalyzed diluted.

17. Determine linear ranges semi-annually. Spike at historical values. Each metal must be recovered at 90-110%.
18. Determine MDLs annually using the requirements of SOP QA-009. MDL's shall be determined more frequently if there is a change to the method or instrumentation that may alter the sensitivity of the measurement. An MDL study is performed for each instrument. MDLs should be less than  $\frac{1}{2}$  the reporting limit, but cannot be higher than the reporting limit.
19. Determine IDLs annually.

#### MAINTENANCE

1. Clean the nebulizer periodically in a 2% Nitric acid solution. Sonication of the nebulizer in this solution is also helpful in removing deposits or particulate matter.
2. Clean the torch periodically by soaking in 2% HNO<sub>3</sub>.
3. Sample pump tubing should be inspected every day and changed when showing signs of wear.
4. Clean sample cone and/or skimmer cone periodically.
5. Check the water level in the chiller.
6. An Agilent service engineer performs scheduled periodic maintenance every six months.
7. All other maintenance shall be documented in the instrument maintenance log. If a particular problem occurs, document the problem and the solution. For additional information on instrument maintenance and documentation requirements see GCAL SOP GEN-012.
8. Refer to the ICPMS operating manual for troubleshooting the instrument. If additional help is still required, contact Agilent Technical Support for assistance in troubleshooting. If the analyst



is still unable to resolve the instrument issue, then Agilent must perform maintenance on the instrument. Any time the analyst troubleshoots the instrument it must be documented in the instrument maintenance log and must include the solution. Refer to GCAL SOP GEN-012 for documentation requirements.

## CALCULATIONS

## 1. Sample Concentration -

$$\text{mg/L or mg/kg analyte in sample} = A \frac{V_f}{V_i} \times d$$

A = mg/L analyte in processed sample

V<sub>f</sub> = final volume of sample (mL)

V<sub>i</sub> = initial volume (mL) or weight (g) of sample

d = dilution factor (if required)

The units may be converted to ug/L or ug/kg

## 2. Matrix spike and Matrix Spike Duplicate Recovery

$$\% \text{ Recovery} = \frac{(\text{SSR} - \text{SR})}{\text{SA}} \times 100$$

SSR = spiked sample result

SR = sample result

SA = spike added

## 3. Matrix Duplicate Relative Percent Difference (RPD)

$$\text{RPD} = \frac{\text{SR} - \text{SDR}}{(\text{SR} + \text{SDR})} \times 100$$

2

SR = Sample result

SDR = sample duplicate result

$\frac{(\text{SR} + \text{SDR})}{2}$  = average of SR and SDR

2

TABLE I

ELEMENT	Selected Isotope	Type	Assigned ISTD
Ag	107	Target	115
Al	27	Target	45
As	75	Target	45
Ba	137	Target	103
Be	9	Target	45

ELEMENT	Selected Isotope	Type	Assigned ISTD
Bi	209	INST	
Ca	44	Target	45
Cd	111	Target	103
Co	59	Target	45
Cr	52	Target	45
Cu	63	Target	45
Fe	57	Target	45
In	115	INST	
K	39	Target	45
Li	6	INST	
Lu	175	INST	
Mg	24	Target	45
Mn	55	Target	45
Mo	95	Target	103
Na	23	Target	45
Ni	60	Target	45
Pb	208	Target	209
Rh	103	INST	
Sb	121	Target	159
Sc	45	INST	
Se	78	Target	45
Sn	120	Target	115
Sr	88	Target	103
Tb	159	INST	
Ti	47	Target	45
Tl	205	Target	209
V	51	Target	45
Zn	66	Target	45

ELEMENT	Selected Isotope	Type	Assigned ISTD
Zr	90	Target	103

## SOFTWARE

## TROUBLESHOOTING

The ICP-MS uses Mass Hunter to control the instrument and perform all data calculations. In the event of software malfunction, please contact Agilent technical support or GCAL's IT Director for troubleshooting help.

METHOD PERFORMANCE  
(METHOD 6020A)

- 1) Method Performance is described in Section 13.0 of SW-846 6020A. This includes descriptions of a multi-laboratory study for aqueous and solid matrices. Results of the study are summarized in the reference method Table 3 and Table 4.

METHOD PERFORMANCE  
(METHOD 200.8)

- 1) Method Performance is described in Section 13.0 of EPA 200.8 Revision 5.4 (EMMC Version). Method performance including typical IDLs, MDLs, method precision and bias is described and detailed in the referenced Tables for total and dissolved metal determinations for each matrix.

POLLUTION PREVENTION See QAPP Section 10.2

## WASTE MANAGEMENT

See SOP GEN-009

## Corrective Actions

Refer to the Quality Control and Data Acceptance Criteria Section of this SOP for procedures on handling QC sample results that fall outside the required acceptance criteria.

For corrective actions other than those specified in this SOP, refer to SOP GEN-018, Non-Conformances/Corrective Actions.